Towards the reliable chemical stability testing of the single base gunpowder using a microcalorimetry method

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Abstract:

Introduction/purpose: Gunpowder is a type of explosive material (EM), a mixture of chemical compounds capable of releasing their potential energy in a very fast exothermic chemical reaction. This paper investigates the single base gunpowder samples.

Methods: Microcalorimetry (MC), or heat flow calorimetry (HFC), is the only modern method that monitors the direct cause of autoignition - the rate of heat release, which is a key factor for gunpowder storage explosive safety. It is based on high-sensitivity calorimeters which allow monitoring of chemical reactions at low speeds. The microcalorimeter "TAM III" was used and the method given by the NATO standard STANAG 4582. A very reliable result was obtained on the chemical stability of the observed single base gunpowder samples, as well as an assessment of its behavior in the next 10 years.

Results: The thermal activity of gunpowder depends on several factors, the most important of which are: chemical composition, size and shape of the gunpowder grain, the degree of decomposition of the gunpowder, storage conditions, etc. Namely, it is a much more exact and consistent indicator of the chemical stability of gunpowder compared to the critical diameter.

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Conclusion: The MC method should be used both for monitoring the chemical stability of gunpowder during storage and for the prediction of the service life of gunpowder.

Key words: single base gunpowder, microcalorimetry, heat flow calorimetry, TAM III, STANAG 4582.

Introduction

Gunpowder is a type of explosive material (EM), a mixture of chemical compounds capable of releasing their potential energy in a very fast exothermic chemical reaction. The process of combustion (deflagration) is the basic form of explosive conversion of gunpowder (Jeremić, 2007), used to launch the projectile in the barrel of the weapon or for propulsion. At the same time, the heat required for the chemical decomposition process is transferred through EM exclusively by thermal conduction, and the burning speed is relatively low and ranges from several millimeters to several tens of centimeters per second. At these rates of explosive conversion, the released gaseous products can be used to eject projectiles from the barrel of weapons or to propel rocket projectiles, which is why gunpowders are often called propellants.

Gunpowder is divided in several ways, and the most common is the division according to the physical state of its components. In this sense, gunpowder is divided into two basic groups (Jeremić, 2007):

1. Homogeneous gunpowder created by gelatinization of nitrocellulose molecules under the influence of an organic solvent or gelatinization agent. With these powders, all components are found in the nitrocellulose molecule; together with it, they form a unit that cannot be separated mechanically.

2. Composite gunpowder is created by mixing crystals of mineral compounds rich in oxygen and a binder, which is always of organic origin, and at the same time is a carrier of combustible materials. This is a pure physical mixture and all components can be easily separated.

Homogeneous powders are further divided, according to the number of energy components in their composition, into (Jeremić, 2007):

- Single base gunpowders, which contain one energy component, nitrocellulose (NC),
- Double base gunpowders, which contain two energy components: NC and glycerin trinitrate (NG), i.e. (sometimes but less often) diethylene glycol dinitrate (DEGDN), and

• Triple base gunpowders, which contain three energy components: NC, NG, and nitroguanidine (NQ).

Composite powders include composite rocket propellants and black powder.

The basic components of homogeneous gunpowder are nitric acid esters: NC, NG (DEGDN), and NQ (Bajić, 2015). These are unstable chemical compounds which are subject to slight thermal decomposition even at ordinary temperatures. Among the products of gunpowder decomposition are nitrogen oxides which cause further autocatalytic exothermic decomposition of nitrogen esters (primarily NC), and thus change the physical-chemical and ballistic properties of gunpowder. Thermal decomposition of nitrogen esters can cause accumulation of heat and an increase in temperature in the mass of gunpowder, as a result of which self-ignition of gunpowder can occur. Historically, self-ignition of gunpowder came immediately after the conquest of the technology of low-smoke gunpowder production. Several accidents happened on warships (de Klerk, 2015).

To slow down the decomposition of nitrogen esters (primarily NC), stabilizers are added to powders. The stabilizers are substances that react very quickly with released nitrogen oxides and the acids that arise from them and absorb free radicals well, thereby preventing their further autocatalytic action. Most widely used stabilizers today are aromatic amines, and diphenylamine (DPA) is most commonly used to stabilize single base gunpowder (Stine, 1991; Grbović & Stojiljković, 2005; Jeremić & Grbović, 2006; Rusly et al, 2024). The decomposition of homogeneous gunpowder is accompanied by a series of phenomena, such as the release of gases, loss of mass, reduction of the average molecular weight of NC, consumption of stabilizers, generation of heat, etc. Regarding this, numerous methods have been developed to monitor the aforementioned changes, determine the current chemical stability, and predict the service life of gunpowder. One of the modern methods is microcalorimetry. This is the only method that measures the direct cause of self-ignition of gunpowder, i.e., the rate of heat release. It is based on calorimeters with high sensitivity, which enables the monitoring of chemical reactions at very low speeds (Grbović & Stojiljković, 2005; Jeremić & Grbović, 2006; Jelisavac et al, 2014).

Microcalorimetry method

Microcalorimetry (MS), or heat flow calorimetry (HFC), is the only modern method that monitors the direct cause of autoignition - the rate of heat release, which is a key factor for gunpowder explosive safety. It is based on high-sensitivity calorimeters, which allow monitoring of chemical reactions at low speeds.

With this method, according to the National Defense Standard (SORS 8069/91), the chemical stability of gunpowder is determined by measuring the rate of heat development in the center of the sample, which is isothermally heated at a constant temperature (SORS, 1991). The measured value of the rate of heat development (thermal activity due to the decomposition of gunpowder) is further used to calculate the critical diameter as a function of temperature. By comparing these values with the diameter of the ammunition case in which the gunpowder is stored, the state of chemical stability is evaluated, that is, the possibility of self-ignition of the gunpowder under certain storage conditions (SORS, 1991). Based on the values and the set criteria, the tested propellants are categorized (Jelisavac et al, 2014). The decision on further action about the propellant storage procedure will be made later. The standard prescribes the microcalorimetry method for determining thermal activity, which is based on the theory of thermal explosion and monitors the amount of released heat as the immediate cause of spontaneous combustion (Jelisavac et al, 2014).

According to STANAG 4582, the rate of heat development is measured at a constant temperature between 60 °C and 90 °C during the calculated time for the selected temperature (STANAG, 2004). This time is considered equivalent to isothermal storage for 10 years at 25 °C. The heat equivalent of all physical and chemical processes that take place in the system with heat exchange is measured, that is, the total heat flow is measured. The maximum permissible heat flow limit, which is also dictated by the experimental temperature represented, is used as a criterion for sufficient chemical stability (STANAG, 2004). The release of heat is of vital importance especially when large quantities of gunpowder are stored, as the heat generation from a given volume of stored gunpowder can become greater than the heat removal when self-heating begins, which can eventually cause self-ignition. This can happen even when the stabilizer is sufficiently present in the gunpowder.

Microcalorimetric measurements are performed with high-sensitivity devices, which can monitor very slow chemical reactions with low thermal activity (1μW/g). The microcalorimetry method is based on the thermal

theory of self-ignition of gunpowder. During the years of application, some shortcomings of this method have been identified (Jeremić & Grbović, 2006, Chelouche et al, 2020). NATO's concept of measuring the thermal activity of propellant is based on the use of far more sophisticated microcalorimetric equipment with high sensitivity for heat flow measurement (Jelisavac et al, 2014).

Experimental part

Materials

Microcalorimetric measurements were performed on several samples of naturally aged single base gunpowder taken mainly from the stored collections. A few samples were taken from the ammunition. The basic compositions of the tested gunpowder are given in Table 1, and their dimensions and shapes in Table 2.

Table 1 – Composition of the tested single base gunpowder samples

Type of gunpowder	Chemical composition %					
	NC	N content	K_2SO_4	DPA	CI	Moisture
1	2	3	4	5	6	7
NC-27	96.80±0.90	≤ 13.10		1.50±0.30		1.35±0.25
NC-29	≤ 94.30	13.05±0.10		1.20±0.30	4.00±0.80	1.25±0.25
NC-37	96.70±0.90	≤ 1.10		1.50±0.20		1.35±0.25
NC-39	93.8-96.0	≤ 13.00		1.00-2.00		1.00-1.60
NC-40		≥ 12.95		≥ 1.20		1.30
NC-44	96.70±0.70	13.20±0.05		1.45±0.15		1.30±0.20
NC-45	85.35-90.40	13.20±0.05	1.0-1.3	0.70-1.30	1.30-1.50	0.80-1.20
NC-123	96.75±0.85	≤ 13.00		1.50±0.20		1.35±0.25
NC-151	48.80±2.00	≤ 13.15	47.0±1.0	1.00±0.20		0.80±0.20
NC-281	86.65±2.55	10.00±1.50		1.55±0.25		1.25±0.25

Table 2 – Dimensions and shapes of the tested single base gunpowder samples

Type of gunpowder	Outer diameter, D (mm)	Channel diameter, d (mm)	Wall thickness, Wa (mm)	Length, L (mm)	The shape of a powder grain
1	2	3	4	5	6
NC-27	1.20±0.15	0.30±0.05	0.45±0.05	6.50±1.00	Single channel cylinder
NC-29	1 .95±0.10	0.33±0.05	0.81±0.05	3.35±0.15	
NC-37	5.60±0.35	0.50±0.10	1.02±0.07	12.05±0.55	

Type of gunpowder	Outer diameter, D (mm)	Channel diameter, d (mm)	Wall thickness, Wa (mm)	Length, L (mm)	The shape of a powder grain
1	2	3	4	5	6
NC-39	3.25-3.35	0.18-0.26	0.60-0.70	5.00-6.00	
NC-40	~3.20	0.20±0.05	0.60-0.75	~6.30	
NC-44	6.00-6.90	0.50-0.70	1.12-1.22	13.50-15.50	
NC-45	1.91-2.10	0.15-0.25	0.85-0.95	3.90-4.10	Like NC-27
NC-123	4.80±0.20	2.50±0.20	1.18±0.08	705.0±0.15	Tube
NC-151	2.10±0.25	0.55±0.10	0.775±0.125	6.50±1.00	Single channel cylinder
NC-281	0.86±0.06	0.20±0.03	0.33±0.03	1.80±0.20	Single channel cylinder

Microcalorimetric measurements were performed on 10 types of single base gunpowder of different chemical compositions and different dimensions and shapes, as well as for different storage conditions.

Method

The samples are crushed as needed and each individual or group is subjected to thermal treatment, during which the data on the thermal activity of the treatment is collected for each of the samples, both to calculate the critical diameter of self-ignition according to the National Defense Standard (SORS, 1991), and to monitor the thermal activity during isothermal testing according to STANAG 4582. Microcalorimetric measurements were performed using the "TAM III" microcalorimeter. (Figure 1).

Figure 1 – Heat flow microcalorimeter TAM III

After grinding and sieving, the gunpowder samples were placed in hermetically sealed vials for sample testing. The sample volume was about 2 cm3. The vials with the samples were set into the device after its calibration. The measurement was carried out at 90 °C and the duration of the test was 3.43 days (82.5 hours) when the released heat from the sample becomes greater than 5 J/g. According to STANAG 4582, the value of the measured heat flow should be lower than the criterion prescribed for the limits of heat flow, 350.0 μW/g at 90 °C (STANAG, 2004; Jelisavac et al, 2014).

Results and the discussion

For the analysis of the previous results according to the MC method, the results of the chemical stability tests of single base gunpowder, which were obtained in the TRZ Kragujevac laboratory, were taken. Thus, all available results for single base gunpowder were analyzed (Table 3), which made it possible to observe the behavior of gunpowder with different compositions and geometric shapes and storage time.

Table 3 – The results of the chemical stability tests for the investigated gunpowder samples

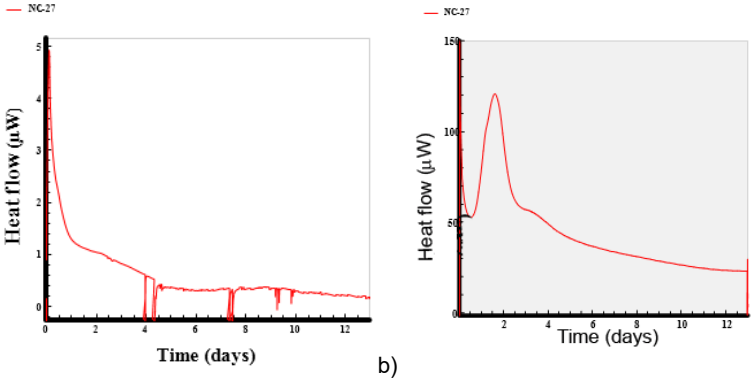
Gunpowder		t_E	Stabilizer content, mas %			100 °C	D_{cr}	P_{max}	Sample source
Type	Lot	(y)	DPA_o	DPA	DPA_{ef}		(m)	(μW/g)	
1	2	3	4	5	6	7	8	9	10
NC-27	МБЛ 8766	15	1.62	1.2	1.46	7.00			PC-2
		18		1.13	1.47	5.00	0.48		
NC-27	МБЛ 8766	19	1.62	0.87	1.33	6.75	q(333)<0		PC-2
		20		0.24	1.11	3.75			
		22		0.06	0.66	0	**<0.30**	**227**	
	МБЛ 0196	8	1.26	1.32	1.44	5.00	0.50	41.4	PC-1
NC-37	МБЛ 8451	17	1.50	0.22	0.86	3.75	0.71		PC-2
		18		0.16	0.92	3.50			
		19		0.18	0.76	3.75	0.53		
		20		0.11	0.86	3.50	q(333)<0		
		21		0.06	0.66	3.50	0.5		
		22		0.1	0.65	4.25			
		25		0.17	0.81	3.75	0.57	59.3	
	МБЛ 8454	17	1.66	0.24	0.77	3.75	0.55		PC-2
		18		0.12	0.86	3.75			
		19		0.18	0.80	3.75	0.52		
		20		0.15	0.81	4.75	0.56		
		21		0.10	0.67	3.50			

Gunpowder		t_E	Stabilizer content, mas %			100 °C	D_{cr}	P_{max}	Sample source
Type	Lot	(y)	DPA_o	DPA	DPA_{ef}		(m)	(μW/g)	
1	2	3	4	5	6	7	8	9	10
NC-37	МБЛ 8454	22	1.66	0.07	0.72	5.25			PC-2
		25		0.14	0.80	3.75	0.77	76.3	
	МБЛ 8464	17	1.70	0.18	1.02	4.75	q(333)<0		PC-2
		18		0.07	0.96	3.75			
		19		0.11	0.9	3.75	0.5		
		20		0.08	0.98	4.00	q(333)<0		
		21		0.03	0.74	3.75			
		22		0.04	0.77	5.25			
		25		0.14	0.87	3.75	0.79	62.1	
	МБЛ 8578	15	1.51	0.25	0.95	4.00			PC-2
		16		0.23	0.94	4.50			
		17		0.18	1.09	3.75			
		18		0.2	0.92	3.75	q(333)<0		
		19		0.16	0.69	4.00	0.53		
		20		0.13	0.74	3.75	0.5		
		21		0.1	0.74	5.12			
		24		0.17	0.82	3.75	0.61	85.6	
	МБЛ 87120	15	1.64	0.44	1.3	5.00	0.5		PC-2
		18		0	0.75	3.75			
		19		0	0.67	4.75			
		22		0.03	0.48	4.50	0.62	20.3	
	МБЛ 94168	15	1.48	0.09	0.63	5.25	0.51	67.9	Ammo
NC-123	MBL 8104	20	1.6	0.17	0.97	4.25			PC-2
		21		0.01	0.76	3.25			
		22		0.08	0.8	3.75	0.53		
		23		0	0.88	3.50	0.65		
NC-123	MBL 8104	24	1.6	0	0.69	3.75	0.53		PC-2
		25		0.01	0.69	5.00			
		26		0.36	0.92	4.00	0.53		
		28		0.08	0.70	4.75	0.48	79.3	
	МБЛ 8408	17	1.67	0.25	0.86	3.75	0.5		PC-2
		18		0.1	0.82	3.75			
		19		0.2	0.82	3.75	0.59		
		20		0.15	0.75	4.75	0.69		
		21		0.09	0.87	3.75	0.68		
		22		0.11	0.76	4.75			
		23		0.20	0.79	3.75	0.68		
		25		0.22	0.79	3.75	0.55	58.9	
NC-29	МБЛ 8830	16	1.25	1.04	1.17	10.00			PC-1
		21		0.98	1.15	5.00	0.62	23.2	
NC-151	МБЛ 9803	7	1.20	0.8	0.8	8.00			PC-1

Gunpowder		t_E	Stabilizer content, mas %			100 °C	D_{cr}	P_{max}	Sample source
Type	Lot	(y)	DPA_o	DPA	DPA_{ef}		(m)	(μW/g)	
1	2	3	4	5	6	7	8	9	10
NC-151	МБЛ 9803	11	1.20	0.82	1.08	5.00	0.47	101.4	PC-1
NC-281	МБЛ 9715	8	1.50	1.25	1.25	8.00			PC-1
		9		1.27	1.4	7.75			
		10		1.08	1.3	8.75			
		12		1.18	1.43	7.00		7.2	
	МБЛ 0623	3	1.37	1.29	1.44	7.00	0.55	24.3	Ammo
NC-40	МБЛ 8921	15	1.41	0.94	1.17	7.75			PC-1
		17		0.58	1.06	5.75			
		18		0.18	1.05	5.75			
		20		0.26	0.77	6.00	0.46	61.3	
NC-44	МБЛ 8801	16	1.42	1.05	1.2	8.00			PC-1
		21		1.06	1.22	8.00	0.65	18.9	
NC-45	МБЛ 8801	16	0.88	2.23	2.37	10.00			PC-1
		21		0.77	0.95	8.00	0.59	15.4	
	МБЛ 9312	17	0.81	2.34	2.47	9.25	q(333)<0		Ammo
		16		0.83	0.98	8.00	0.48	28.1	
NC-39	МБЛ 7401	29	1.38	0.29	0.97	7.00	0.48		PC-2
		30		0.27	0.85	7.00	0.4		
		31		0.23	0.74	6.00			
		32		0.18	0.87	7.00	0.52		
		35		0.24	0.89	7.00	0.51	84.20	

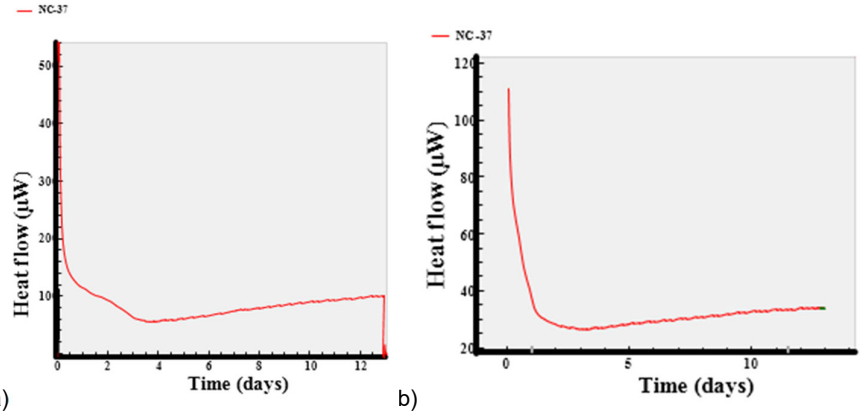
In Table 3, t_E is the gunpowder service time in years, DPA_0, DPA and DPA_{eff} represent initial, measured and effective content of the stabilizer respectively, 100 °C stands for the results of 100 °C heating method, D_{cr} is the critical diameter, and P_{max} is the maximum heat flow. The sample source stands for the source of gunpowder samples, propellant collection one or two (PC-1 or PC-2) and the samples taken from the ammunition (Ammo).

Using the "TAM III" microcalorimeter, thermal activity was measured on a total of 16 lots of 10 different types of single base gunpowder (Figures 2-10). Based on the obtained results, the maximum heat flow was determined according to STANAG 4582. In addition, for each lot of gunpowder that was tested according to the MC method, the results were compared with the results of the chemical stability data obtained according to the HPLC and 100 °C methods, as well as the analysis of all previous results of testing the chemical stability of those powders. This made it possible to comprehensively analyze the results obtained by the MC method.



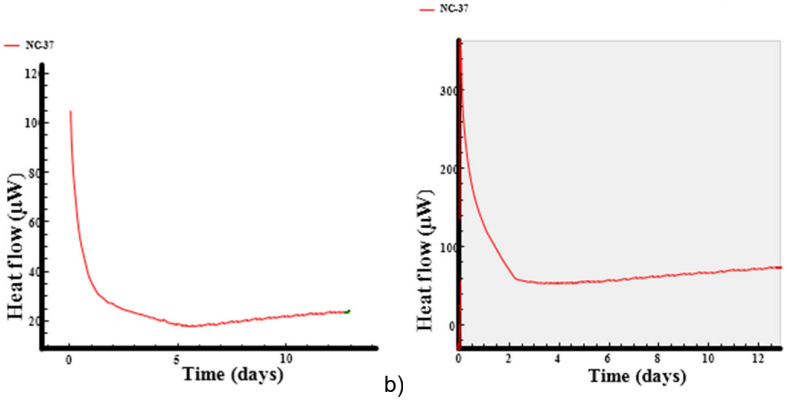
a) b)

Figure 2 – Heat flow diagrams for NC-27 a) sample 1 and b) sample 2 gunpowder



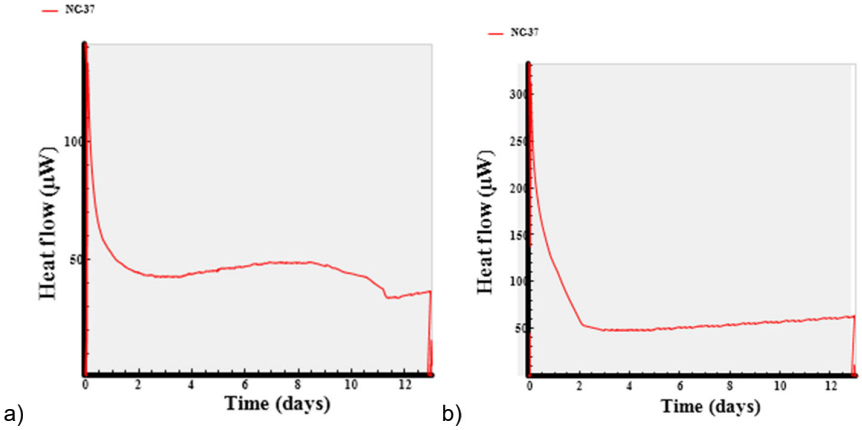
a) b)

Figure 3 – Heat flow diagrams for NC-37 a) sample 1 and b) sample 2 gunpowder



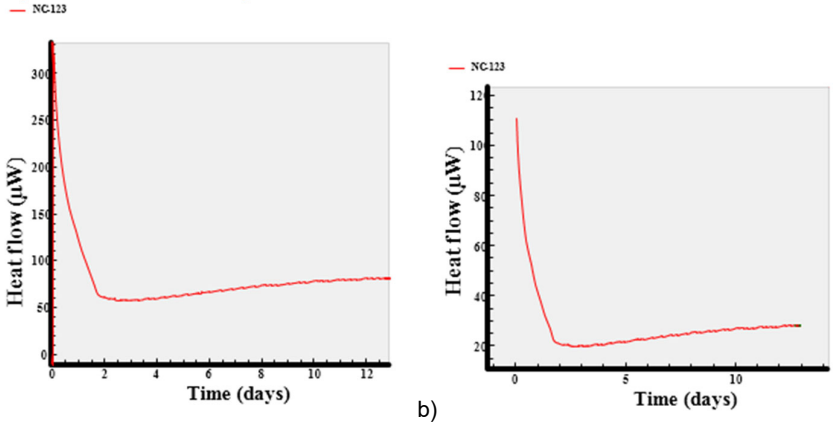
a) b)

Figure 4 – Heat flow diagrams for NC-37 a) sample 3 and b) sample 4 gunpowder



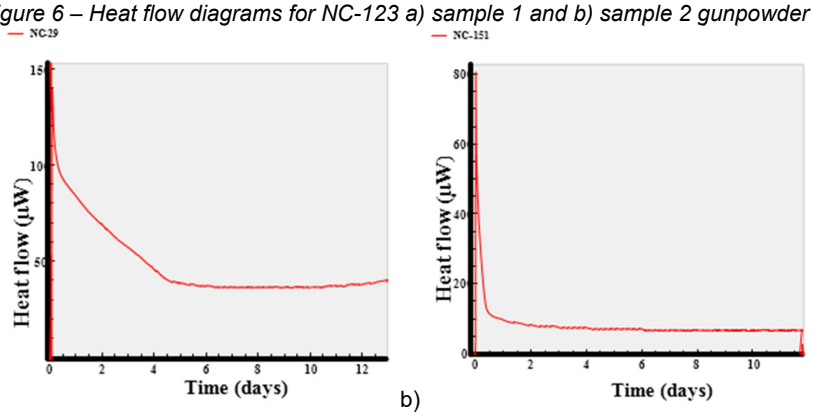
a) b)

Figure 5 – Heat flow diagrams for NC-37 a) sample 5 and b) sample 6 gunpowder



a) b)

Figure 6 – Heat flow diagrams for NC-123 a) sample 1 and b) sample 2 gunpowder



a) b)

Figure 7 – Heat flow diagrams for a) NC-29 and b) NC-151 gunpowder

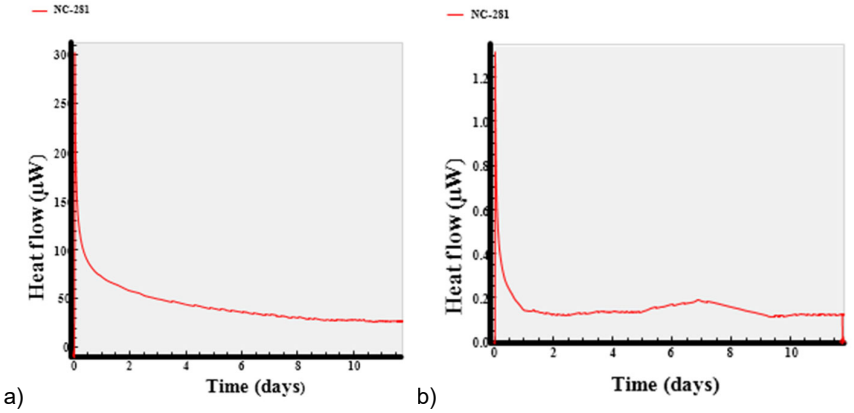


Figure 8 – Heat flow diagrams for NC-281 a) sample 1 and b) sample 2 gunpowder

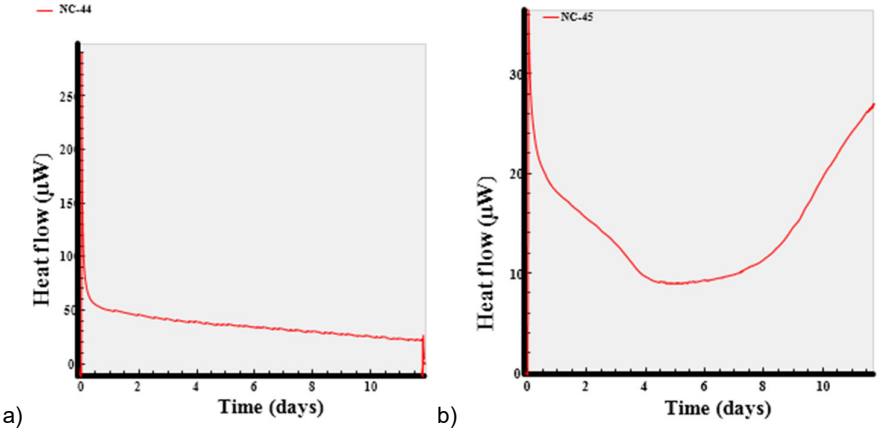


Figure 9 – Heat flow diagrams for a) NC-44 and b) NC-45 gunpowder

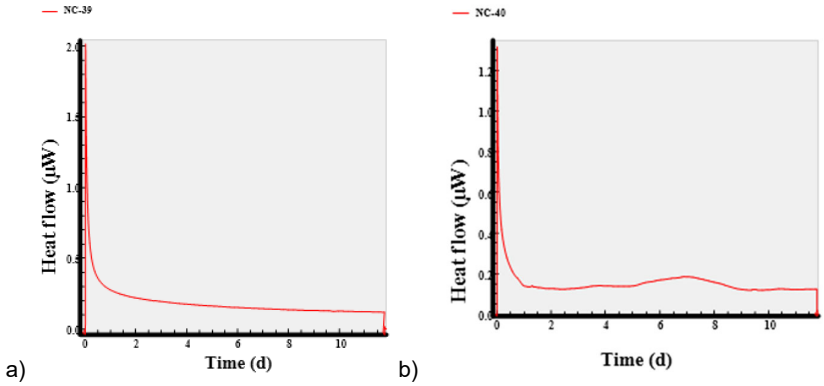


Figure 10 – Heat flow diagrams for a) NC-39 and b) NC-40 gunpowder

Microcalorimetric measurements were performed on single base gunpowders because they are the most common in Serbian Armed Forces ammunition. An analysis of the previous results of determining the critical diameter according to SORS 8069/91 was performed. These results do not correlate with the results of the HPLC method and the 100 ºC heating method. It happens that with a decrease in the stabilizer content (that is, an increase in the number of days at 100 ºC), the critical diameter remains unchanged or increases. In addition, the dependence of the critical diameter on other factors that significantly affect the chemical stability of gunpowder, such as the chemical composition and the dimensions and shape of the gunpowder grain, cannot be observed. It was agreed that this method is imprecise, which can be explained by several reasons (inaccuracy of the device, the short heating time of the sample about the measurement temperature, the critical diameter is recalculated with the introduction of a series of approximations, etc.) and that it would be very justified to apply the MC method prescribed by NATO standard STANAG 4582. In practice so far, not a single gunpowder has been assessed as unstable based on this criterion, while based on the results of the HPLC method and the 100 ºC heating method, many lots of gunpowders have been assessed as unstable and destroyed. For example: powder NC-27, sample 1, is stable according to the MC method prescribed by standard SORS 8069/91, while according to the HPLC method as well as according to the 100 ºC heating method, this powder is unstable. Given that SORS 8069/91 stipulates that the thermal activity monitoring test is in the second place of evaluation (behind the stabilizer content monitoring method), this gunpowder was assessed as unstable. At the same time, this gunpowder does not satisfy the given criterion according to the MC method prescribed by NATO standard STANAG 4582. This example confirms the shortcomings of the first method and the precision of the second MC method.

Based on the MC measurements performed using the "TAM III" device, it was concluded that the thermal activity of gunpowder depends on several factors, the most important of which are: chemical composition, size and shape of the gunpowder grain, the degree of decomposition of the gunpowder, storage conditions, etc. (the gunpowder charge was not analyzed because gunpowder samples and gunpowder collections were taken). At the same time, the maximum heat flow - Pmax (criterion according to STANAG 4582 standard) is a much more exact and consistent indicator of the chemical stability of gunpowder compared to the critical diameter - Dc (criterion according to SORS 8069/91 standard). Namely, even measurements using a modern "TAM III" microcalorimeter

cannot provide a reliable assessment of the critical diameter. The reason for this lies in a whole series of assumptions that are introduced in the assessment of the possibility of exchanging the generated heat with the environment, the conditions for heat accumulation, and the theory of self-ignition, i.e., under which critical conditions the possibility of self-ignition occurs. In this sense, the measurement of thermal activity about the determination of the critical diameter of self-ignition is a far more reliable method because it is based on the measurement and evaluation of the exact parameter of decomposition - heat flow, while when determining the critical diameter of self-ignition, the values of individual parameters are theoretically obtained and insufficiently confirmed.

The maximum heat flow depends a lot on the chemical composition of the gunpowder. The extremely good chemical stability of NC-29 and NC-45 gunpowder is the result of a favorable composition. Namely, in addition to DPA (stabilizer), these powders also contain CI (Centralite I) as a plasticizer or ballistic modifier. During long-term heating at an elevated temperature, as the DPA concentration decreases, CI is also included in the reaction with nitrogen oxides, even though it was not added to the gunpowder composition as a stabilizer. At the same time, the high thermal activity of NC-151 gunpowder can be explained by its unfavorable chemical composition. Namely, this gunpowder contains about 47% of K_2SO_4, a hygroscopic compound that releases moisture during microcalorimetric measurement, which leads to the formation of nitrous and nitric acid in the gunpowder, which autocatalytically accelerates NC decomposition and increases heat generation.

The maximum heat flow depends on the dimensions and shapes of the powder grain. Among the examined powders, from this point of view, the most characteristic is the NC-281 powder. Namely, due to its small dimensions and a favorable shape, this gunpowder exchanges the generated heat very quickly, which results in a small heat flow.

The maximum heat flow depends very much on the quality of the basic raw materials used for the production of gunpowder. The powders that were produced during the period of sanctions (e.g. NC-37 sample 6, and NC-45 sample) show a higher thermal activity compared to other younger lots of these types of powders. The fact is that many lots of NC-27 and NC-37 powders, which were produced during the 1990s, showed significantly lower chemical stability according to the HPLC method and the 100 ºC heating method compared to older lots of these types of powders. This can be explained by the lower quality of the basic raw materials.

The thermal activity of gunpowder also depends on the degree of NC decomposition. However, from this aspect, the results of different lots of

the same type of gunpowder can be compared. It is observed that older lots of gunpowder with a higher degree of decomposition generate more heat than the lots with a lower degree of decomposition. This is confirmed by the results of two lots of three types of gunpowder (NC-27, NC-37, and NC-123). At the same time, this does not mean that samples of different types of gunpowder, which according to the HPLC method have approximately the same stability, will also have similar results according to the MC method. On the contrary, the results often vary widely.

Conclusion

By analyzing the results of the determination of the critical diameter of self-ignition of gunpowder according to SORS 8069/91 standard, it was concluded that they do not correlate with the results of the HPLC method and the 100 ºC method. In addition, the dependence of the critical diameter on other factors that significantly influence the chemical stability of gunpowder, such as the chemical composition and the dimensions and shapes of the gunpowder grain, cannot be observed. It has been concluded that this method is imprecise, which can be explained by several reasons (inaccuracy of the device, the short heating time of the sample about the measurement temperature, the critical diameter is recalculated with the introduction of a series of approximations, etc.).

Measurements using a microcalorimeter "TAM III" and the method given by NATO standard STANAG 4582, gave reliable results on the current chemical stability of gunpowder, as well as an assessment of its behavior in the next 10 years. At the same time, the results of this MC method are in good correlation with the results of the HPLC and 100 ºC methods. In addition, by applying this method, the dependence of the thermal activity of gunpowder on the factors that most significantly affect the chemical stability of gunpowder, such as chemical composition, size and shape of gunpowder grains, degree of decomposition of gunpowder, quality of basic raw materials, storage conditions, etc., can be observed.

The proposal is to apply the MC method given by NATO standard STANAG 4582 both for monitoring the chemical stability of gunpowder during storage and predicting the service life of gunpowder, as well as for the quality control of newly produced gunpowder.

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Hacia la prueba fiable de estabilidad química de la pólvora de base única mediante un método de microcalorimetría

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CAMPO: tecnología química
TIPO DE ARTÍCULO: artículo científico original

Resumen:

Introducción/propósito: La pólvora es un tipo de material explosivo (EM), una mezcla de compuestos químicos capaces de liberar su energía potencial en una reacción química exotérmica muy rápida. Este artículo investiga las muestras de pólvora de base única.

Métodos: La microcalorimetría (MC), o calorimetría de flujo de calor (HFC), es el único método moderno que controla la causa directa de la autoignición- la tasa de liberación de calor, que es un factor clave para la seguridad de los explosivos en el almacenamiento de pólvora. Se basa en calorímetros de alta sensibilidad que permiten monitorear reacciones químicas a bajas velocidades. Se utilizó el microcalorímetro "TAM III" y el método dado por la norma OTAN STANAG 4582. Se obtuvo un resultado muy confiable sobre la estabilidad química de las muestras de pólvora mono-base observadas, así como una evaluación de su comportamiento en los próximos 10 años.

Resultados: La actividad térmica de la pólvora depende de varios factores, los más importantes son: composición química, tamaño y forma del grano de pólvora, grado de descomposición de la pólvora, condiciones de almacenamiento, etc. Es decir, es un indicador mucho más exacto y consistente de la estabilidad química de la pólvora en comparación con el diámetro crítico.

Conclusión: El método MC debe usarse tanto para monitorear la estabilidad química de la pólvora durante el almacenamiento como para predecir la vida útil de la pólvora.

Palabras claves: pólvora de base única, microcalorimetría, calorimetría de flujo de calor, TAM III, STANAG 4582.

Подход к надежному способу определения химической стойкости одноосновных порохов методом микрокалориметрии

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РУБРИКА ГРНТИ: 61.43.00 Технология взрывчатых веществ и средств химической защиты
ВИД СТАТЬИ: оригинальная научная статья

Резюме:

Введение/цель: Порох − это разновидность взрывчатого вещества, представляющая собой смесь химических соединений, способных высвобождать свою потенциальную энергию в результате быстрой экзотермической химической реакции. В данной статье рассматриваются образцы одноосновного пороха.

Методы: Микрокалориметрия или калориметрия теплового потока является единственным современным методом, который следит за главной причиной самовоспламенения − скоростью тепловыделения. Скорость тепловыделения является ключевым фактором взрывобезопасности при хранении пороха. Метод основан на высокочувствительных калориметрах, которые позволяют отслеживать химические реакции на низких скоростях. В данном исследовании использовался микрокалориметр "TAM III", метод которого соответствует стандарту НАТО STANAG 4582. В ходе исследования получен надежный результат по химической стойкости испытуемых образцов одноосновного пороха, а также оценка его поведения в течение следующих 10 лет.

Результаты: Термическая активность пороха зависит от нескольких факторов, наиболее важными из которых являются: химический состав, размер и форма порохового зерна, степень разложения пороха, условия хранения и т.д. А это гораздо более точные и последовательные показатели химической стойкости, чем критический диаметр.

Выводы: Метод микрокалориметрии следует использовать как для контроля химической стойкости пороха при хранении, так и для прогнозирования срока хранения пороха.

Ключевые слова: одноосновный порох, микрокалориметрия, калориметрия теплового потока, TAM III, STANAG 4582.

Приступ поузданом начину одређивања хемијске стабилности једнобазних барута применом методе микрокалориметрије

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ОБЛАСТ: хемијске технологије
КАТЕГОРИЈА (ТИП) ЧЛАНКА: оригинални научни рад

Сажетак:

Увод/циљ: Барути представљају експлозивне материје, смешу хемијских једињења способних да своју потенцијалну енергију ослободе након врло брзих егзотермних хемијских реакција. У овом раду испитују се узорци једнобазних барута.

Методе: Микрокалориметрија, или калориметрија топлотног флукса, једина је савремена метода која прати директан узрок самозапаљења – брзину ослобађања топлоте, која представља кључни фактор пиротехничке безбедности током складиштења. Заснива се на високоосетљивим калориметрима који омогућавају праћење хемијских реакција при малим брзинама. У раду је коришћен микрокалориметар „TAM III“ и метода која је наведена у стандарду „STANAG 4582“. Веома поуздани резултати добијени су за хемијску стабилност испитиваних узорака једнобазних барута, као и оцена њиховог понашања у наредних 10 година.

Резултати: Топлотна активност барута зависи од неколико фактора, од којих су најважнији: хемијски састав, димензије и облик барутног зрна, степен деградације барута, услови складиштења и др. Она представља егзактнији и конзистентнији индикатор хемијске стабилности него што је то критични пречник.

Закључак: Метод микрокалориметрије треба користити како при праћењу хемијске стабилности барута тако и при предвиђању животног века употребе барута.

Кључне речи: једнобазни барут, микрокалориметрија, калориметрија топлотног флукса, TAM III, STANAG 4582.

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