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Comparative Examination of the Chemical Stability of Powders and Double – Base Rocket Propellants by Measuring Heat Activities and Stabilizer Content

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The problem of assessing the chemical stability of nitrocellulose (NC) based propellants is examined with a focus on a comparative analysis of the home country based methodology and modern methods used in the world. The measurements were performed using the instrumental methods such as microcalorimetry and high pressure liquid chromatography. During many years of implementation, the disadvantages of the domestic method of microcalorimetry for the chemical stability assessmentby measuring the critical diameter of NC propellants in accordance with SORS 8069/91 standard have been noticed. Therefore, the priority of the research in the field of thermal activity of NC propellants is given to the method of measuring the heat flow in accordance with STANAG 4582 NATO standard. The analysis of the results of the assessment of the chemical stability of different types of gunpowders (GP) stored in collections under continental and mediterranean climate conditions has shown that there is no correlation between the results of the heat flow measurements done in accordance with STANAG 4582 and the results of the measurements of the critical diameter and stabilizer content in naturally aged GP done in accordance with SORS 8069/91. There is agreement between the results of the chemical stability assessment by measuring heat flow in accordance with STANAG 4582 and the results of the measurements of the stabilizer content in artificially aged NC propellants done in accordance with AOP-48 Ed.2.

Key words: **gunpowder, double-base rocket propellant, chemical stability, microcalorimetry, heat flow, liquid chromatography, stabilizers.**

Used simbols

AOP – Allied Ordnance Publication;
CI – Ethyl centralite;
DPA – Diphenylamine;
DPA eff – Diphenylamine effective
GP – Gunpowder;
HFC – Heat Flow Microcalorimetry;
HPLC – High Performance Liquid Chromatography;
NATO – North Atlantic Treaty Organization;
NC – Nitrocellulose;
SORS – Standard odbrane Republike Srbije;
STANAG – Standardisation Agreement of NATO/PfP.

Introduction

DURING the storage of ammunition charged by NC propellants, series of chemical-physical processes that cause their aging occur [1]. The chemical aging process takes place under very complex mechanisms and, under certain conditions, it can cause the change of the characteristics of propellants below acceptable, marginal values which guarantee the security of ammunition in use and storage. Storage of ammunition charged by propellants of reduced quality becomes risky because of a possibility of spontaneous self-ignition and large scale accidents. The danger of spontaneous self-ignition during storage is a consequence of the fact that NC is subjected to a slow but constant thermal decomposition. Among the products of decomposition of NC, which is the basic energy component of homogeneous propellants, there is an increase of nitrogen oxides content which causes its further autocatalytic exothermic degradation. Under certain, critical, conditions, this can lead to spontaneous self-ignition of propellants in ammunition [2].

Many years of research, throughout the world, have resulted in more modern instrumental methods for monitoring changes in the physico-chemical properties of propellants caused by ageing [3]. Today, during the control of chemical stability, the method of microcalorimetry and the method of determination of stabilizer content have priority [4].

Stabilizer content is a parameter closely connected to the process of thermal decomposition of NC. It is measured by the methods of high performance liquid chromatography (HPLC), gas chromatography, thin layer chromatography and UV-Vis spectrophotometry [3]. The thermal properties of propellants are measured by the heat flow microcalorimetry method [3, 4].

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Assessment of the stability of propellants in accordance with SORS 8069/91, STANAG 4582 and AOP-48 Ed.2 standards

The thermal activity monitoring is one of the main parameters on the basis of which the stability of propellants can be assessed [5]. The reactions of the thermal decomposition of nitrocellulose are exothermal. It can lead to the accumulation of heat in propellants the further consequence of which may be an increase in temperature to such an extent that it creates the conditions for propellant spontaneous self-ignition. The microcalorimetric method for assessing the stability of NC propellants is standardized by SORS 8069/91 [6]. It prescribes the determination of the critical diameter for spontaneous self-ignition of propellants. Based on its value and in accordance with the criteria set forth, the categorization of the tested propellants is carried out [6]. A decision on a further action related to the procedure of propellant storage is made subsequently. The standard prescribes the method of microcalorimetry to determine thermal activity, which is based on the theory of thermal explosion and monitors the amount of heat released as an immediate cause of spontaneous self-ignition [6]. The microcalorimetry method, an original national method, was introduced twenty years ago. This method includes the measurement of the heat generation rate in the most critical part of the sample (geometric center) during isothermal heating at a temperature of 60°C. The critical diameter (D_c) is the diameter of the sample catridge or ammunition caliber at which heat generated by thermal decomposition of propellants equals the amount of heat that can be exchanged with the surroundings in such conditions [4, 6, 7]. If the diameter of an examined propellant sample is larger than the critical one, this leads to an autocatalytic process. In that case the rate of heat generation is greater than the rate of heat removal. The consequence can be self-ignition of NC propellants [4, 6, 7]. During the years of implementation, some disadvantages of the mentioned method have been identified [4, 8]. NATO's concept of measuring the thermal activity of propellants is based on the use of far more sophisticated high sensitivity microcalorimetric equipment for heat flow measuring.

STANAG 4582 prescribes the procedure for assessments of chemical stability of propellants based on the Heat Flow Microcalorimetry (HFC) method of measuring heat flow at elevated temperatures. This method has the ability to predict chemical stability for at least 10 years of storage at an isothermal storage temperature of 25°C [5, 9].

The standard prescribes time-temperature test conditions by equation (1), giving the period during which the sample would be exposed to the influence of heat equivalent to the influence of heat during isothermal storage of 10 years at 25ºC [9].

$$t_m = t_{25} e^{\frac{E_1\left(\frac{1}{T_m}-\frac{1}{T_{60}}\right)+E_2\left(\frac{1}{T_{60}}-\frac{1}{T_{25}}\right)}{R}} \quad (1)$$

where:

t_m - time of measurement at T_m, days;

t_{25} - time of storage at T_{25}, 10 years = 3652.5 days;

E_1 - activation energy for heat generation caused by thermal decomposition of propellants at temperatures higher than 60°C, J/mol; E_1 = 120 kJ/mol;

T_m - temperature of measurement, K;

T_{60} - temperature of change of the activation energy for NC thermal decomposition, 60°C = 333.15 K;

T_{25} - temperature of storage, 25°C= 298.15 K;

R - universal gas constant, R= 8.314 J/K mol;

E_2 - activation energy for heat generation caused by thermal decomposition of propellants in a temperature range from 25°C to 60°C, J/mol, E_2 = 80 kJ/mol.

The time of the test duration (t_m) in the microcalorimeter [9], and the criteria limit of heat flow values measured by HFC method depend on the selected test temperature T_m, and are given in Table 1 [9].

Table 1. The values of the test time and the limits of the heat flow P_1 at different test temperatures [9].

T_m [°C]	t_m [day]	P_1 [μW/g]
60	123.0	9.8
70	34.8	34.5
80	10.60	114.0
90	3.43	350.0

The activation energy of the reaction of stabilizer consumption during single temperature isotermal heating of different types of NC propellants is approximately equal to the value of the activation energy obtained by testing the same samples in the microcalorimeter. Therefore, AOP-48 Ed.2 and STANAG 4582 standards use the same test conditions given in equation (1) related to the test temperature and time [9-12]. Chemical stability of propellants with different stabilizers such as: diphenylamine (DPA), ethyl centralite (CI), methyl centralite (CII), acardite II, 2-nitro-DPA, etc., can be assesse by these procedures [9, 10]. NC based propellants will be assessed according to the following criteria [10]:

Criterion 1: Maximum decrease in effective stabilizer during ageing (in % of the initial level) ≤ 80 %.

Criterion 1: Minimum percentage of effective stabilizer remaining after ageing ≥ 0.2 %.

Experiment

The gunpowders (GPs) stored under conditions of continental climate (collection marked as KB-1) and under those of mediterranean climate (collection marked as KB-2), were investigated.

The chemical stability of the mentioned GPs was assessed in accordance with:

- SORS 8069/91 by measuring the remained stabilizer content in naturally aged GPs from the collections as well as the critical diameters of GPs [4, 6, 13];
- STANAG 4582 by measuring the heat flow of GPs [4, 9].

Also, different types of propellants were artificially aged [4] in the heating tubes within a thermal block at 90°C (Fig.1). Then, the content of the remaining stabilizer in these isothermal heated samples was determined periodically by high pressure liquid chromatography [4].

Figure 1. Isothermal heating of NC propellants

The stabilizer content in the tested propellants was determined by the procedures described in [4,14] using the liquid chromatograph "EF Waters 1525 Binary HPLC pump" with a thermostat for column heating, the manual injector "Rheodine Model 7125", and the photodiode array detector "Waters 2998 PDA" (Fig.2).

Figure 2. The liquid chromatograph "Waters"

The heat flow measurement of the same tested NC propellants was performed in accordance with STANAG 4582 on the HFC microcalorimeter TAM III (Fig.3).

Figure 3. Heat flow microcalorimeter TAM III

After grinding and sieving, the propellant samples were placed in hermetically sailed vials for sample testing [4, 9]. The sample volume was about 2 cm^3. 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 from the time when the realised heat had become greater than the limit value, 5 J/g (Table 1). To satisfy the requirements for propellant stability, the value of the measured heat flux should be lower than the criterion prescribed for the limits of heat flux, 350.0 μW/g at 90°C (Table 1).

The critical diameters of self ignition of naturally aged NC GPs from KB-1 and KB-2 were measured in accordance with SORS 8069/91 in the microcalorimeter MICRO III of domestic production [4, 6]. After grinding and weighing, the GP samples were placed in a cylindrical brass vessel (4 cm in diameter and 4 cm high) and tempered first at 60°C and then at 70°C. The heat released at each of the temperatures was measured.

Results and Discussion

Correlation of the measurements of heat flow and stabilizer depletion in isothermal heated propellants

In order to determine the mentioned correlations, heat flow measurements were performed in accordance with STANAG 4582 and stabilizer content measurements in accordance with AOP-48 Ed.2., by heating propellants at 90°C [4]. The results of the measurements of heat flow of (3.43 days at 90°C) in the HFC microcalorimeter are shown in Figures 4-10.

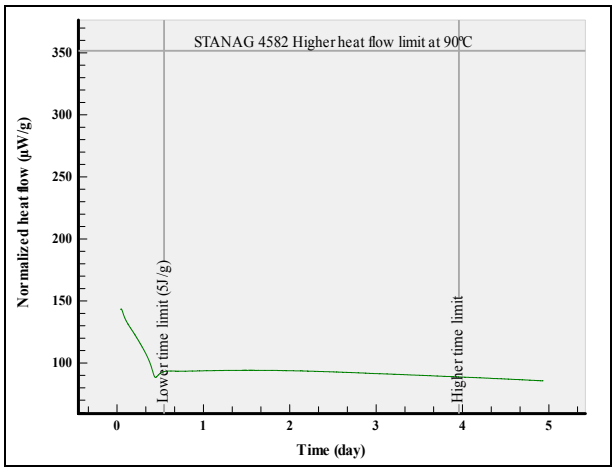

Figure 4. HFC (3.43 days at 90°C) NGR 316, 2005

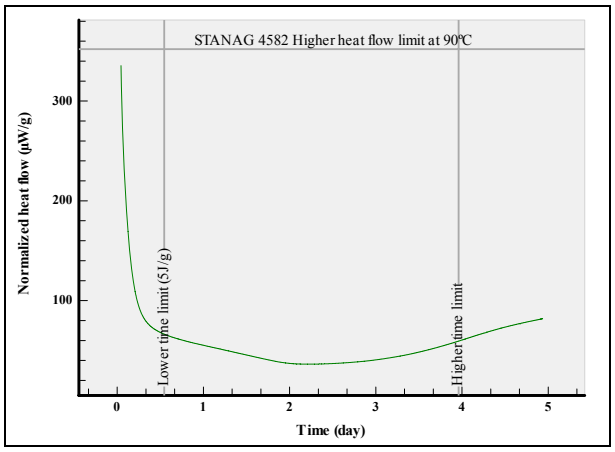

Figure 5. HFC (3.43 days at 90°C) NC-40, S 9226

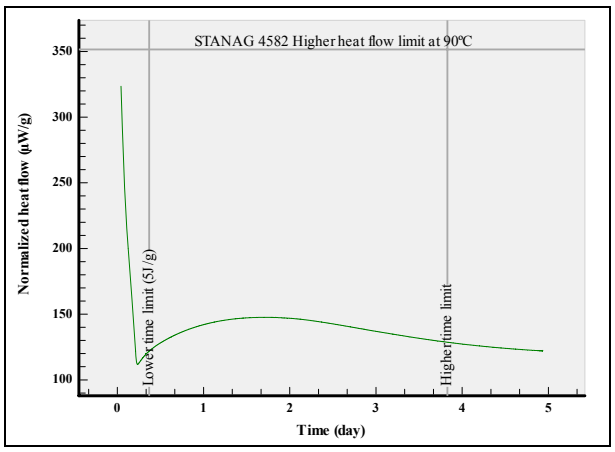

Figure 6. HFC (3.43 days at 90°C) KUB-M, 1977

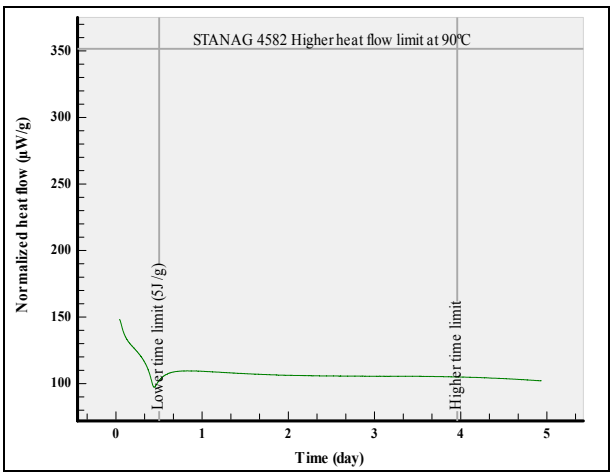

Figure 7. HFC (3.43 days at 90°C) RS1-60, 1975

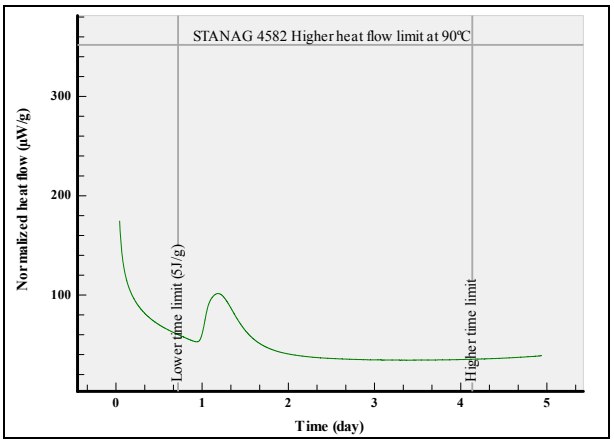

Figure 8. HFC (3.43 days at 90 °C) NC-48, 2011

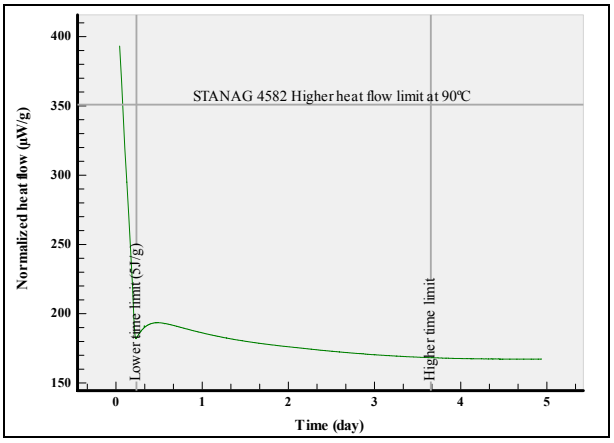

Figure 9. HFC (3.43 days at 90 °C) BR-PZ-AM-2, 1983

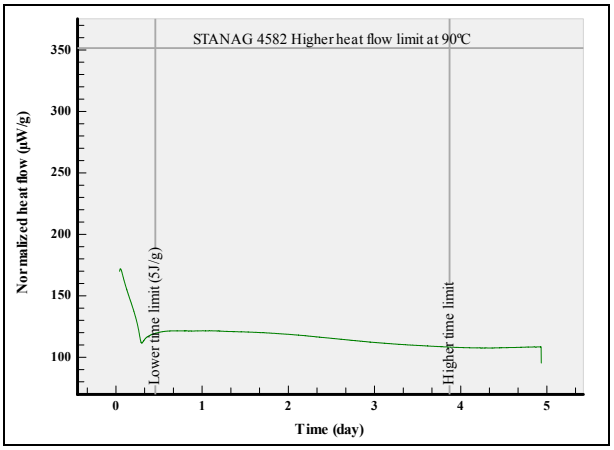

Figure 10. HFC (3.43 days at 90 °C) NGR-176, 2005

The results of the assessment of the chemical stability of the five double base rocket propellants and two single base GPs examined by the method of heat flow measurement in accordance with STANAG 4582 and by the method of stabilizer depletion in accordance with AOP-48 Ed. 2. are shown in Table 2 [4]. The heat flow values marked with * are the average values of two measurements [4].

Table 2. Assessment of the chemical stability of propellants, methods based on isothermal heating at a temperature of 90ºC

GP and DB RP	STANAG 4582		AOP-48		
	Heat flow, µW/g	HFC criteria	$D_{DPA\ eff}$, D_{CI}, D_{CII}, %	DPA eff CI, CII, mas %	Criteria of stabilizer content
KUB-M, 1977.	115.8*	≤ 350 µW/g ⇩ ≥10 years at 25 ºC	38.78	0.78	$D_{DPA\ eff}$, D_{CI}, D_{CII} ≤ 80 % and DPA $_{eff}$, CI, CII ≥0,2 mas % ⇩ ≥ 10 years at 25 ºC
BR-1 PZ AM, 1983.	132.6*		37.06	1.80	
RS-1-60, 1975.	102.2*		24.43	2.08	
NGR-176, 2005.	108.5		26.71	2.25	
NGR-316, 2005.	84.3		24.46	2.10	
NC-40 S 9226	61.8		20.76	0.84	
NC-48, 2011.	36.9*		-	-	-

The analysis of the results showed that all tested propellants satisfy the limit criteria of stability set out in STANAG 4582 and AOP-48 standards. Therefore, NATO standards predict and guarantee that tested propellants will remain chemically stable if stored at ambient temperatures (25ºC) for a minimum of 10 years. The results of the chemical stability assessment by the method of heat flow measurement and by the method of stabilizer determination overlapped completely. [4]. An excelent compatibility of the results of the two leading NATO methods of assessment of the chemical stability of propellants, based on the same principle of accelerated aging samples at single temperature, was confirmed. If necessary, the priority is given to the HFC method, which is a more direct method related to measuring the consumption of stabilizers, because it measures a direct cause of spontaneous self ignition.

Examination of the stabilizer content - heat flow correlation during the same temperature-time conditions of isothermal heating

The best way to examine the stabilizer content - heat flow correlation, could be by occasionally interruption of the experiment in the microcalorimeter and then the measurement of residual stabilizer in the propellant. In the absence of the results of such experiments and in order to study the different phases of the process of thermal decomposition of propellants, this research is based on the measurements, the results of which are shown in Fig.11.

The figure simultaneously shows the results of:

- periodical measurement of the stabilizer contents in the samples of single base powder NC-40, isothermally heated in a thermo-block for 5 days at 90°C [4], and
- continuous measurement of the heat flow of the same sample, NC-40, during the experiment in the HFC microcalorimeter, 5 days at 90°C, in accordance with STANAG 4582 [4].

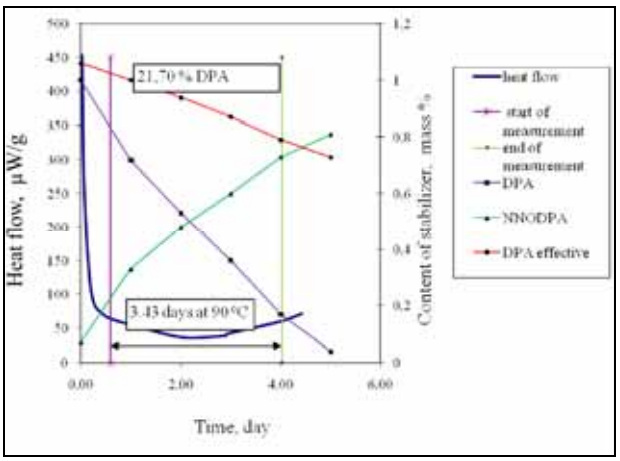

Figure 11. Heat flow and stabilizer content, NC 40 S 9226 at 90ºC

By analysing Fig.11, we can explain the interdependence of the stabilizer consumption during the period of continuously measured heat flow during the thermal decomposition of NC-40 powder [15]. The initial part of the HFC curve with the maximum value of heat flow around 454 μW/g is a consequence of the primary reactions of powder NC-40 with residual oxygen in the HFC vial. The integral part of the area under the curve is in correlation with the stabilizer content of DPA stabilizer and the amount of oxygen present in the atmosphere [16]. After decreasing the part of the HFC curve, the following part of the curve is the result of a constant rate of decomposition of the tested single-base powder, which is why about the same amount of heat is released during this period of measurements.

In accordance with the mechanism of the chemical transformation of DPA and its derivatives [4] of single-base GPs, after the phase decomposition during which all the DPA was consumed, its nitroso derivative N-nitroso-DPA (NNODPA) takes the role of a stabilizer in single-base gun powder. Although at the end of the experiment only about 0.17 mass % of the DPA remained in the propellant, STANAG 4582 test guaranteed chemical stability in the period of 10 years at 25°C. This figure clearly shows that the NNODFA takes the role of the stabilizer. The content of NNODFA in NC-40 at the end of the experiment was 0.73 mass %. The measured heat flow is 60.5 μW/g during the test by the HFC method (3.43 days at 90°C). Under the same conditions of isothermal heating of NC-40 (3.43 days at 90°C), the relative consumption of effective DPA is D_{DPAeff} = 21.70 % and it is equivalent to the amount of the stabilizer that would be consumed in the period of storage of 10 years at 25ºC.

Correlation of the results of the measurements of heat flow and stabilizer content in naturally aged gunpowder

The test results of the chemical stability assessment of naturally aged gunpowder were analysed [4]. Table 3 shows the average values of the chemical stability parameters of gunpowder measurements [4].

Table 3. Average values of the chemical stability parameters of GPs from collections KB-1 and KB-2

Measured parameter	KB-1	KB-2
$D_{DPA\ eff}$, mas %	19.36	24.58
Heat flow, μW/g	107.75	203.72
Critical diameter, m	0.46	0.35

The analysis of the *average* values of the measurement results showed that the GPs samples from the collection stored in the conditions of mediterranean climate, KB-2, showed a faster decreasing stability compared to the gunpowder samples from the collection stored in the continental climate conditions, KB-1. It results in: increase in relative consumption of effective DPA (D_{DFAeff}) and heat flow and decrease in the critical diameter. These facts are the consequences of the mediterranean extreme climate conditions in the GPs collection marked as KB-2 (higher temperature, humidity and salty atmosphere).

Fig.12 shows the comparison of the results of the measurements of the relative consumption of the stabilizer, the heat flow and the critical diameter of single-base GP samples: NC-45, NC-123, NC-18, NC-281, NC-37, NC-43 and NC-44, which were sampled from KB-1 collection [4].

The observation of each GP *individually*, despite the trend of increasing of a relative consumption of diphenylamine, and effective heat flow, there are some deviations, which mostly depend on the GP age and chemical composition, the geometry of the propellant grain and the technological process of GPs production.

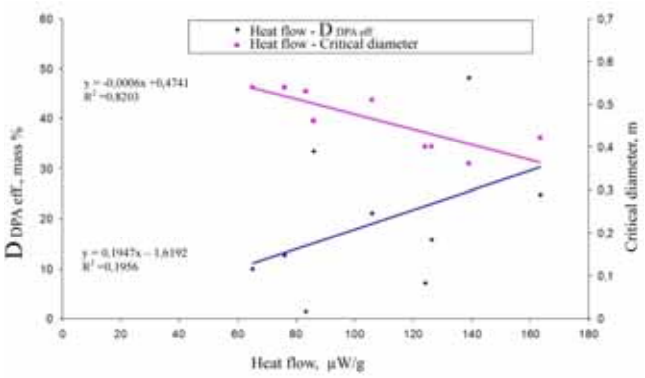

Figure 12. Correlation of the measurements of the heart flow, the stabilizer consumption and the critical diameter of GP from KB-1

Table 4 presents an interesting case of NC-281 single-base GP, whose two series, 9715 and 9816, were stored 12 years (t_E) under the same conditions of mediterranean climate (collection KB-2). The mentioned series of gunpowder NC-281 have the same compositions, geometries, moisture content and the same content of remained DPA (1.3 mass%) and effective DPA (1.48 mass%) [4].

Table 4. Assessment of the chemical stability of NC-281 from KB-2 [4]

NC-281 series	t_E, years	Stabilizer content, mas %						STANAG 4582 Heat flow [μW/g]
		DPA_o	DPA	NNO DPA	2NDPA	4NDPA	(SORS 8069/91) DPA eff	
9715	12	1.37	1.3	0.04	0.11	0.03	1.48	22.2
9816	12	1.40	1.3	0.03	0.12	0.03	1.48	180.9

The content of DPA and its derivatives was determined by HPLC in accordance with SORS 8069/91 and it provides information on the current state of naturally aged gunpowdersand their current chemical stability [4]. Both tested series of NC-281 single-base GP have the same content of DPA and DPAeff. It indicates their equivalent current chemical stability at the time of the measurement of stabilizer content.

However, two series of the mentioned NC-281 GP have drastically different mesasured values of heat flow (22.2 μW/g and 180.9 μW/g). The higher value of heat flow, 180.9 μW/g, indicates greater thermal activity of NC-281 S 9816 due to faster thermal decomposition when it is heated to 90°C during the experiment.

Isothermal heating is in the basis of the process of measuring heat flow, which is why the method accordance to

STANAG 4582 has the ability to predict the behavior of GPs. Therefore, a higher heat flow value (180.9 μW/g) indicates and predicts a faster dynamics of aging of 9816 series in the subsequent period of storage. Considering the same composition, geometry, moisture content and storage conditions, the differences in the dynamics of aging of two series of the same NC-281 single-base GP can be explained by the quality of used nitrocellulose and the technological process of NC-281 production. This example generally illustrates the advantage of the procedure of assessment of chemical stability of propellants based on isothermal heating related to the procedure without isothermal heating [4].

Correlation of the results of the measurements of the heat flow and the critical diameter of gunpowder

The results of the measurements of the heat flow and the critical diameter in GPs, sampled from the collection with continental climate, KB-1, are compared in Fig.12. The observation of each GP *individually*, despite the trend of increasing of effective heat flow, and decreasing of critical diameter of GPs, there are some deviations. This can be explained by the disadvantages of the domestic method of determining the critical diameter such as: the introduction of a series of approximations, imprecision of the MICRO III calorimeter of domestic production and the mode of measurement, low reproducibility, no correlation with other methods, the device retaining heat poorly, and the rate of heat evolution measured indirectly through the temperature difference, ΔT, with a problem of the precision of the ΔT determination [2].

The difference of the method of measuring heat flow in accordance with STANAG 4582 related to the SORS 8069/91 method of determining the critical diameter is reflected in much greater precision and reproducibility of measurement, a small number of samples, higher productivity due to a large number of samples to be tested at once and ease of interpretation of the results [2]. For these reasons, in order to improve the assessment of the chemical stability of NC propellants, it is proposed to introduce the method of measuring heat flow in accordance with STANAG 4582 instead of the method of measuring the critical diameter in accordance with SORS 8069/91 standard.

Conclusion

The paper deals with a comparative analysis of the results of assessing chemical stability of different naturally and acceleratingly aged propellants using the national and world methodology. The results were obtained by measuring the heat flow in the HFC microcalorimeter, measuring the critical diameter in the microcalorimeter of domestic production and determining the stabilizer content by the HPLC method.

The analysis of the *average values* of the measurements of chemical stability parameters of NC gunpowders, showed that the GPs from the collection stored in the conditions of mediterranean climate, KB-2, had a faster decreasing stability compared to the gunpowder samples from the collection stored in the continental climate conditions, KB-1. Increasing of heat flow and relative consumption of effective DPA and decreasing of the critical diameter of examined GPs are the consequences of the mediterranean extreme climate conditions in the GPs collection marked as KB-2 (higher temperature, humidity and salty atmosphere).

However, in *individual* cases there are deviations which can be explained by the perceived disadvantages of the SORS 8069/91 microcalorimetry method and a substantially different principle of assessing chemical stability in accordance with STANAG 4582 method compared to SORS 8069/91 method for determining the stabilizer content. The correlation between the method for assessing chemical stability by measuring heat flow (STANAG 4582) and the method of determining the stabilizer content in naturally aged gunpowders (SORS 8069/91) is not confirmed but there is a compatibility of STANAG 4582 method with the method of determining the stabilizer content in artificially aged propellants (AOP-48 Ed.2) . It was experimentally confirmed that all tested propellants meet the limit criteria of chemical stability prescribed by STANAG 4582 and AOP- 48 Ed.2 standards and, therefore, sufficient chemical stability under conditions of storage during 10 years at 25°C can be guaranteed. An excelent compatibility of the results of the two leading NATO methods of assessment of the chemical stability of propellants, based on the same principle of accelerated aging samples at single temperature, was confirmed..

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Komparativno ispitivanje hemijske stabilnosti baruta i dvobaznih raketnih goriva merenjem toplotne aktivnosti i sadržaja stabilizatora

Istraživana je problematika praćenja hemijske stabilnosti baruta i dvobaznih raketnih goriva (DRG) sa težištem na komparativnoj analizi domaće metodologije i savremenih metoda koje se koriste u svetu. Merenja su izvršena primenom instrumentalnih metoda, kao što su mikrokalorimetrija i tečna hromatografija visokog pritiska. Višegodišnjom primenom, uočeni su nedostaci domaće metode ocene hemijske stabilnosti merenjem kritičnog prečnika baruta i DRG prema standardu SORS 8069/91. Stoga je prioritet pri istraživanju u oblasti toplotne aktivnosti baruta i DRG dat metodi merenja toplotnog fluksa prema NATO standardu STANAG 4582. Analiza rezultata ocene hemijske stabilnosti različitih vrsta NC baruta čuvanih u kolekcijama u uslovima kontinentalne i mediteranske klime pokazala je da ne postoji korelacija rezultata merenja toplotnog fluksa prema standardu STANAG 4582 sa rezultatima merenja kritičnog prečnika i sadržaja stabilizatora u prirodno starenim barutima, prema SORS 8069/91. Utvrđena je usaglašenost rezultata ocene hemijske stabilnosti merenjem toplotnog fluksa prema STANAG 4582 i rezultata merenja sadržaja stabilizatora u ubrzano starenim barutima i DRG prema AOP-48 Ed.2.

Ključne reči: **barut, dvobazno raketno gorivo, hemijska stabilnost, mikrokalorimetrija, toplotni fluks, tečna hromatografija, stabilizator.**

Сравнительное изучение химической устойчивости порошковых и дважды базовых ракетных топлив путём измерения тепловой активности и содержания стабилизатора

Мы исследовали проблему исследования химической устойчивости порошковых и дважды базовых топлив (ДБТ) с акцентом на сравнительном анализе домашних методологий и современных методов, используемых в мире. Измерения проводились с использованием инструментальных методов, таких как микрокалориметрия и жидкостная хроматография высокого давления. На протяжении нескольких лет реализации предполагаемых методов замечены недостатки домашнего метода химической устойчивости путём измерения критического диаметра порошка и ДБТ в соответствии со стандартом SORS 8069/91. Таким образом, приоритет исследований в области термической активности порошка и ДБТ обеспечивает способ измерения потока тепла в соответствии с НАТО-стандартом STANAG 4582. Анализ результатов оценки химической устойчивости различных видов НЦ порошка, которые хранятся в коллекциях в условиях континентальных и средиземноморских климатов, показал, что нет никакой корреляции между результатами измерений теплового потока в соответствии со стандартом STANAG 4582 с результатами измерений критического диаметра и содержания стабилизатора в натуральных престарелых порошках в соответствии с SORS 8069/91. Установлено соответствие результатов оценки химической устойчивости путём измерения потока тепла по отношению к стандарту STANAG 4582 и результатов измерения содержания стабилизатора в искусственно престарелых порошках и ДБТ по AOP-48 Ed.2.

Ключевые слова: **Порошок, дважды базовое ракетное топливо, химическая устойчивость, микрокалориметрия, тепловой поток, жидкостная хроматография, стабилизаторы.**

Examen comparatif de la stabilité chimique du poudre et des propergols bibasiques par mesurage de l'activité thermique et du contenu des stabilisateurs

Les problèmes de la suivie de stabilité thermique de la poudre et des propergols bibasiques (DRG) avec le centre d'intérêt sur l'analyse comparative de la méthodologie nationale et les méthodes modernes utilisées mondialement ont été étudiés dans ce travail. Les mesurages ont été effectués à l'aide des méthodes instrumentales telles que microcalorimétrie et chromatographie de haute pression. Après l'utilisation pendant plusieurs ans on a constaté les inconvénients de la méthode nationale pour l'estimation de la stabilité chimique par mesurage du diamètre critique chez la poudre et DGR selon la norme SORS 8069/ 91. Pour cette raison on a donné la priorité dans l'examen de l'activité thermique de la poudre et DRG à la méthode de mesurage du flux thermique selon la norme STANAG 4582 de l'OTAN. L'analyse des résultats de l'estimation de la stabilité chimique chez les différents types de poudre NC gardées dans les collections en conditions climatiques continentales et méditerrranéennes a démontré qu'il n'y avait pas de corrélation entre les résultats de mesurage du flux thermique selon la norme STANAG 4582 et les résultats de mesurages du diamètre critique et le contenu de stabilisateur dans les poudres vieillies artificiellement selon la norme SORS 8059/ 91. On a constaté l'accord des résultats de l'estimation de la stabilité chimique par mesurage du flux thermique selon la norme STANAG 4582 et les résultats de mesurages du contenu de stabilisateur dans les poudres vieillies artificiellement et DRG selon AOP-48 Ed.2.

Mots clés: **poudre, propergol bibasique, stabilité chimique, microcalorimétrie, flux thermique, chromatographie liquide, stabilisateurs.**