

Differential Scanning Calorimetry and Vacuum Stability Test as Methods to Determine Explosives Compatibility

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During production, storage and manipulation of explosive materials it is important to be able to evaluate potential risks when they come in contact with each other or with other non explosives materials. For this reason various analytical and instrumental methods are being developed and implemented to study the chemical compatibility of explosives with other materials. In this research a possibility has been examined of application of thermal method-Differential Scanning Calorimetry (DSC) and Vacuum Stability Test (VST) as methods to determine the compatibility of often used explosive materials: Octogen (HMX), Pentrite (PETN) and Ammonium Perchlorate (AP) with also often used polymer materials: polyamide 12 (PA 12), Hydroxyl Terminated Polybutadiene (HTPB), fluoroelastomer (Viton A). Standard STANAG 4147 was used as criteria to estimate the compatibility between the observed materials.

Key words: energetic materials, explosives, polymers, compatibility, differential scanning calorimetry, vacuum stability test.

Introduction

An important starting point for quality and safe production and storage, and proper handling and disposal is chemical analysis of energetic materials. Additionally, the main goal of chemical analyzing is to develop safer new products for military and non-military application [1, 2]. During the decade, a range of different analyzing methods, procedures and tests have been developed for characterization of the energetic materials. Among the numerous methods, thermal methods can be considered as methods which provide important information about behavior of energetic materials. Although the energetic materials such as propellants, pyrotechnics and explosives are thermodynamically unstable, the thermal methods can be used to predict life time and for choosing an adequate storage condition. They can be also employed for the determination of the compatibility between materials [2, 3].

In realistic conditions energetic materials are rarely used without contact with other materials. Main purpose of compatibility tests is to provide evidence that material can be used in an item of ammunition without disturb to the safety [2, 4, 5]. Compatibility between the energetic material and the other materials can be defined as reaction between them which did not occur after long storage periods, at various conditions which is an ideal case. For practical reasons, contact materials are considered compatible if during and after a specific storage period the functionality and the safety of the components are still acceptable.

On the other hand, incompatibility between the energetic material and the other components may accelerate the aging and impairing the safety and functionality of the entire system. Therefore, precise investigation of stability and

compatibility of energetic materials should be carried out before manufacturing their mixtures and use in technical applications [5].

The energetic materials such as Octogen (HMX), Pentrite (PETN) and Ammonium Perchlorate (AP) are important compounds which are usually incorporated in the polymer materials, forming the Plastic Bonded eXplosives (PBX). The military use of explosives demands a high destruction power, but it has to be safe and easy to handle as well as stock-piled for long periods of time, even in adverse climatic conditions.

STANAG Standard 4147 [6] is the relevant standard which describes the testing and assessment of chemical compatibility for explosives. Although this standard describes several tests, there are two test methods among them that are most often applied: Vacuum stability test (VST) and Differential scanning calorimetry (DSC).

In the former Czechoslovakia, a manometric method was developed to study the thermal stability mainly of propellants [7, 1] known under the name of STABIL: it can be considered, to a certain extent, as an automated variant of the American vacuum stability test [8]. Originally, this procedure was designed for technological quality checking of explosive products and also for the testing of chemical compatibility. Determinations of compatibility by using VST, according to standard, were based on comparison of the volume of evolved gas of pure samples of the observed materials and the volume of evolved gas of their mixture during 40 hours at 100 °C.

The thermal method such as Differential scanning calorimetry has also found application in energetic materials examinations [9]. This method is a thermodynamic-tool for direct monitoring of the changes of phase transitions and for assessment of the heat energy uptake, which occurs in a

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sample within a regulated increase or decrease in temperature [10]. In the explosives field, the differential scanning calorimetry method is the most commonly used for identification of explosive substances over characteristic peaks (endothermic peaks corresponding to the melting point or characteristic phase transitions), calculation of kinetic parameters, and determination of the compatibility of explosive materials with other materials [10-12]. In the range of determining the compatibility of explosive materials with other materials by using DSC technique, according to STANAG Standard 4147 [6], measurements were based on comparison of the obtained peaks from pure samples and peaks from mixtures [2].

This paper describes the compatibility studies between explosives: HMX, PETN and AP and polymer materials: polyamide 12 (PA 12), Hydroxyl Terminated Polybutadiene (HTPB), fluoroelastomer (Viton A) by using thermal method-Differential Scanning Calorimetry and Vacuum Stability Test.

Materials and methods

Materials

In this examination three explosive materials were used, Fig.1., with characteristics given in Table 1:

- Octogen (HMX), cyclotetramethylene-tetranitramine, purity above 95%;
- Pentrite (PETN), Pentaerythritol tetranitrate and, purity above 95% and
- Ammonium Perchlorate (AP), purity above 95.5%, with average particle size 6-10 μm .

Following tests materials were selected, with characteristics given in Table 2:

- Nylon 12 (polyamide 12, PA 12) Nylon 12 - poly(ω -dodecanamide)
- Hydroxyl Terminated Polybutadiene (HTPB), and
- fluoroelastomer (Viton A).

Table 1. Main properties of examined explosives [13-16]

Explosive	HMX	PETN	AP
Density, g/cm^3	1.91	1.77	1.95
Detonation velocity, m/s	9120	8300	6300
Heat of formation, kJ/mol	252.8	-1683.0	-290.7
Melting point, $^{\circ}\text{C}$	204	275	Exothermic decomposition before melting at $>200^{\circ}\text{C}$
Autoignition temperature, $^{\circ}\text{C}$	229	240	240

Table 2. Properties of examined polymer materials [17]

Polymer	Viton A	PA 12	HTPB
Density, g/cm^3	1.82	0.97 – 1.03	
Glass transition temp., $^{\circ}\text{C}$		37 – 43, max operating temp. 70	-90
Viscosity Pa-s	Mooney Viscosity, ML 1+10 at 100°C : 59-71		
Melting temp. range, $^{\circ}\text{C}$		190 – 200 178 – 180	
Solvents	Soluble in low molecular weight ketones, esters		

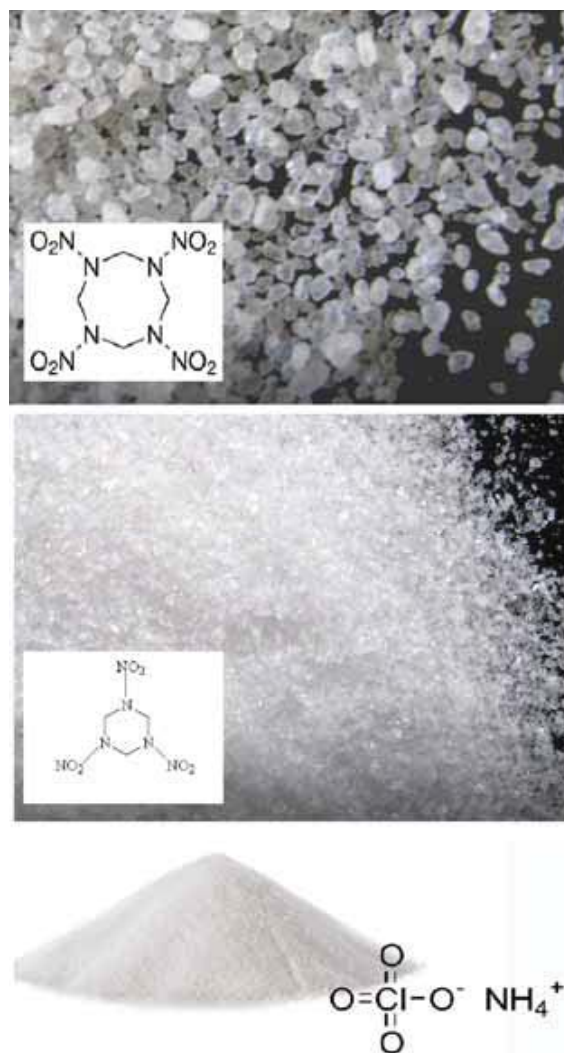


Figure 1. From top to bottom: crystals of HMX, PETN and AP, with their chemical structures

The labels of their mixtures are given in Table 3.

Table 3. Mixture of explosive materials and polymers

Mixture	Name
HMX/Viton A	H-I
HMX/PA 12	H-II
HMX/HTPB	H-III
PETN/Viton A	P-I
PETN/PA 12	P-II
PETN/HTPB	P-III
AP/ Viton A	A-I
AP/PA 12	A-II
AP/HTPB	A-III

The compatibility of explosive materials and polymers in mixtures was determined by the methods of DSC and VST.

Methods

Differential scanning calorimetry (DSC)

Determinations of compatibility were performed by using the DSC Q20, TA Instruments. The instrument was calibrated with Indium at heating speed of 2°C/min and the tests were carried out under nitrogen atmosphere, with the flow rate of 50 mL/min. Explosive and polymers were combined in a 1:1 ratio. Individual sample of explosives and polymers were directly measured, in a range of 1 to 1.5 mg, in proper system (appropriate pan and adequate lid made from aluminum). For the mixture, measurement was the same. At the same system, in order to be in contact, the explosives were placed together with the polymers. The heating rate was 2°C/min in a temperature range from 25 to 320°C for the samples with HMX and 300°C for the samples with PETN and AP.

Vacuum stability test (VST)

Vacuum stability test was carried out on the STABIL 20, OZM Research, by using the STANAG 4147-Procedure B [6]. Mass of 2.5 g of explosives and the same mass of polymers were measured. For the mixture, 2.5 g of the explosives and 2.5 g of the polymers were measured together in the same pot and mix. All the samples were placed into tubes with sensitive electronic pressure transducers. Vacuum stability test possess acquisition unit which processed signals from a barometric pressure transducers and temperature sensor which measured ambient pressure and also temperature during the examination. These ambient data were used for calculation of a volume of evolved gases from each sample under the examination. When the vacuum was achieved entire system was placed into an aluminum heating block and heated for 40 hours at 100°C. The results represent an average value of three measurements.

Results and discussion

The method of differential scanning calorimetry

The application of thermal techniques to the study of explosives required specific considerations due to their mode of operation. Generally, sample size is kept to a minimum for safety reasons since the high rate of energy released during decomposition can lead to "thermal runaway". The study of compatibility by using the DSC method, according to standard [6], consisted of observing changes when an explosive and polymer materials are put together in defined thermal conditions which can affect or/and change the chemical stability. If changes are not present, that indicates compatibility of examined materials in defined conditions observed by DSC test. The conditions of this test are actually simulating the aging of the explosive with an aim to guarantee the safety in its handling, storage and use.

Namely, changes are observed and examined in the field of shift in the peak temperature in the single exothermal corresponding to the decomposition of the explosives. The shift in this peak temperature indicated an interaction between explosive and test materials-polymers.

Figures 2, 3 and 4 presented the DSC thermographs of the samples of explosive materials, polymer materials and their mixtures.

The DSC curve, Fig.2, thermogram for AP had an endothermic temperature peak from 242.09 to 243.58°C, which was ascribed to the transition from the orthorhombic to the cubic phase which occurred at about 240°C [18, 19]. Second pick represents exothermic peak and described temperature decomposition of AP, which occurs below

approximately 300°C [20]. According to Picture 1, the temperature of decomposition was expected and in the range from 265.03 to 275.70°C. The illustrated exothermic pick for pure AP has two maximum peaks. This second unexpected peak probably indicates presence of some impurity in the examined AP sample. Also, the same behavior of two maximum peaks was observed in the examined mixture. So, for analyzing the compatibility by using the DSC method the first decomposition temperature – first peak was chosen as relevant.

Comparing the other DSC curves for the mixture, with curve for pure AP the same endothermic peaks existed, but the values of exothermic peak maximums are in a slight decline. It means that shift in the peak toward a lower temperature, indicated that the presence of the polymers has accelerated the decomposition of explosive.

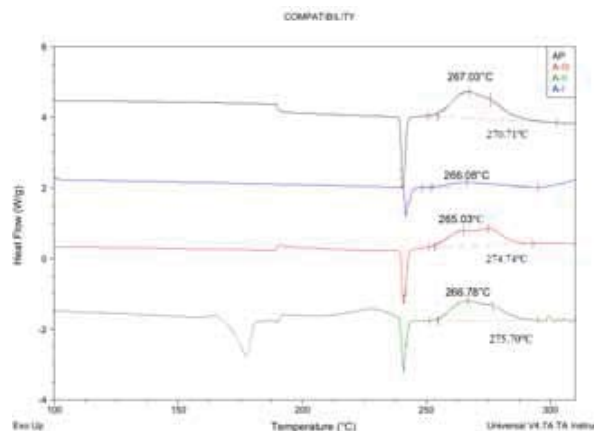


Figure 2. The DSC thermograms of pure AP and mixtures A-I, A-II and A-III

Analyzing the DSC curve for pure HMX, Figure 3, first small endothermic peak in the range from 170 to 190°C represents the crystalline transition from β -HMX to the δ -HMX form [12, 21]. The exothermic peak at the temperature of 276.27°C indicates the decomposition of HMX [21]. Comparing the others DSC curves for the mixture, with curve for pure HMX the same endothermic peaks existed. The same trend, as in the results for AP, continues: the values of exothermic peak, shift in the decomposition peak, maximums are also in a slight decline related to the pure HMX sample.

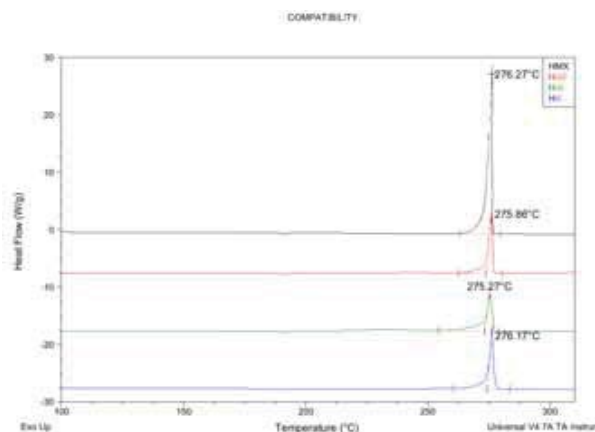


Figure 3. The DSC thermograms of pure HMX and mixtures H-I, H-II and H-III

Fig.4 represents the DSC curves for PETN and their mixture. On the DSC thermogram for pure PETN one endothermic peak is present, i.e. peak of melting point at

141.30°C. The exothermic peak is observed on the temperature of 192.42°C which indicates the decomposition of PETN. Comparisons of the DSC curves for mixture indicate that the value of exothermic peak maximums is decreased. Precisely the shift in the analyzed peaks is caused by presence of polymers which causes earlier decomposition of explosive materials.

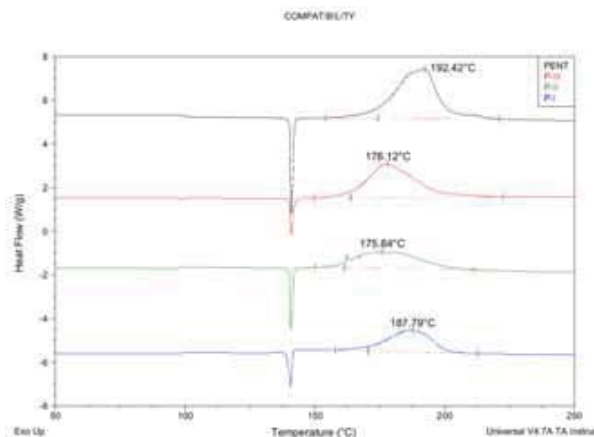


Figure 4. The DSC thermograms of pure PETN and mixtures P-I, P-II and P-III

The temperature of peak maximum was determined and the maximum value of difference in peak temperatures ΔT was calculated by using Thermal Stability Kinetics Analysis for explosives and mixture of explosives and tests materials.

The criteria of compatibility for this method are based on the value of difference in peak temperatures ΔT , and the opinion is formed according to the following:

- $\Delta T < 4^\circ\text{C}$ - the mixture is considered to be compatible
 $4^\circ\text{C} < \Delta T < 20^\circ\text{C}$ - the mixture is considered "moderately" incompatible
 $\Delta T > 20^\circ\text{C}$ - the mixture is considered to be incompatible

The values of maximum peak (T_{max}), the maximum value of difference in peak temperatures (ΔT) and compatibility assessment are showed in Table 4, Table 5 and Table 6.

Table 4. Results of the DSC method for pure HMX and HMX with polymers

Sample	Point of exothermic peak, T_{max} , °C	ΔT , °C	Compatibility assessment
HMX	276.27		
H-I	276.17	0.10	compatible
H-II	275.27	1.00	compatible
H-III	275.86	0.41	compatible

Table 5. Results of the DSC method for pure PETN and PETN with polymers

Sample	Point of exothermic peak, T_{max} , °C	ΔT , °C	Compatibility assessment
PETN	192.42		
P-I	187.79	4.63	compatible
P-II	175.84	16.58	"moderately" incompatible
P-III	178.12	14.30	"moderately" incompatible

Table 6. Results of the DSC method for pure AP and AP with polymers

Sample	Point of exothermic peak, T_{max} , °C	ΔT , °C	Compatibility assessment
AP	267.03		
A-I	266.08	0.95	compatible
A-II	265.03	2.00	compatible
A-III	266.78	0.25	compatible

According to the criteria and the results, samples of mixtures of explosives (HMX and AP) with polymer material are considered as compatible, Table 4 and 6. Analyzing the value of difference in peak temperatures for PETN and their mixture it is obvious that results for samples P-II and P-III are 16.58°C and 14.30°C, respectively, and they are put into the context as "moderately" incompatible, Table 3. When the shifts in the peak temperature of the examined mixture are in between 4°C and 20°C, below the original peak temperatures of explosives, that indicates the degree of incompatibility. This indicates that it is necessary to include other methods to determine the compatibility. Furthermore, applying other method may provide better understanding of compatibility problem and what is more important enable to give an opinion about compatibility for the samples P-II and P-III.

Method of vacuum stability test

The results obtained from method of vacuum stability test are based on the calculation according to the equation (1). The equation presents results of volumes of evolved gas receive from the beginning and from the end (before and after the heating) of experimental for examined samples [7, 8, 22]. Actually, the volumes of evolved gas are calculated on the standard pressure and temperature (SPT), according to the following:

$$V = \left(V_c + V_t - \sum \frac{m_i}{\rho_i} \right) \cdot \left(\frac{p_2 \cdot 273}{273 + t_2} - \frac{p_1 \cdot 273}{273 + t_1} \right) \cdot \frac{1}{1,013} \quad (1)$$

Parameters in the equation (1) are:

- V - the evolved gas volume, cm^3
 V_c - the volume of transducer, cm^3
 V_t - the volume of glass test tubes, cm^3
 m_i - mass of all examination samples, g
 ρ_i - density of all examination samples, g/cm^3
 p_1 - pressure at the beginning of experiment, bar
 p_2 - pressure at the end of experiment, bar
 t_1 - temperature at the beginning of experiment, °C
 t_2 - temperature at the beginning of experiment, °C.

The results of volumes of evolved gas for each: pure explosives (HMX, AP, PETN), pure polymer materials (HTPB, PA 12, Viton A), as well as for their mixtures can give the final value of volume which represents enacted reaction of the compounds. Eq. (2) was used for calculation the volume of evolved gas provoked as a reaction of the compounds in a mixture:

$$V_R = M - (E + S) \quad (2)$$

where:

- V_R - the evolved gas volume produced as a reaction of the compounds in a mixture;
 M - the evolved gas volume of the mixture of energetic and polymer materials, mixed in a mass ratio (2.5 + 2.5) g, (cm^3 at SPT);
 E - the evolved gas volume of the energetic material, mass of the samples 2.5 g, (cm^3 at SPT);

S - the evolved gas volume of the examination material (polymer material), mass of the samples 2.5 g, (cm^3 at SPT).

The criterions of compatibility for this method are represented as calculated volume, V_R (equation 2), and the opinion of compatibility is formed according to the following:

- $V_R < 5 \text{ cm}^3$ - the mixture is considered to be compatible
- $V_R > 5 \text{ cm}^3$ - the mixture is considered to be incompatible
- $V_R = 5 \text{ cm}^3$ - it is necessary to apply other methods of determining the compatibility

The results of volumes of evolved gases measured at 100°C for 40 hours for all samples (HMX, AP, PETN, HTPB, PA 12, Viton A) and their mixtures (Table 1.), as well as the calculated values are presented in Table 7, 8 and 9.

Determinations of compatibility by using VST, according to the defined criteria, were based on comparison of the volume of evolved gas of pure samples and the volume of evolved gas of mixture during 40 hours at 100°C [8].

Table 7. Results of the evolved gas volume for HMX and polymers by method of vacuum stability test

Samples	The evolved gas volume, cm^3			V_R , cm^3
	M	E	S	
H-I	0.802			0.124
HMX		0.133		
Viton A			0.793	
H-II	0.702			0.177
HMX		0.133		
PA 12			0.746	
H-III	0.463			0.010
HMX		0.133		
HTPB			0.320	

The results represented in Table 7, show that HMX is compatible with all tested materials. The opinion about compatibility of HMX and polymers using method of VST agrees with the obtained values from DSC. According to that, there is no need for including other methods for determination the compatibility for this type of explosive material and the observed polymers.

Table 8. Results of the evolved gas volume for PETN and polymers by method of vacuum stability

Samples	The evolved gas volume, cm^3			V_R , cm^3
	M	E	S	
P-I	18.810			0.416
PETN		17.601		
Viton A			0.793	
P-II	1.007			17.340
PETN		17.601		
PA 12			0.746	
P-III	0.551			17.370
PETN		17.601		
HTPB			0.320	

In Table 8, the calculated result for the mixture P-I indicated that value is in the range of criteria for the method of VST. This result confirms compatibility between PETN and Viton A. Further, results for the mixtures P-II and P-III presents the largest increase in volume of evolved gas (17.340 and 17.370, respectively). Putting the obtained results in the

formulation for an opinion of compatibility indicates that the retest is recommended to confirm the results.

Retesting involves applying another method based on a different principle as confirmation. If the second test confirms the incompatibility than the test material is not compatible with examined explosive.

On the other hand, previous method - DSC method is established on changes in the decomposition peak temperature when an explosive and polymer materials are put together in defined thermal conditions. According to the DSC method the obtained results for the same mixtures (P-II and P-III) were in a form as "moderately" incompatible. That fact directly indicates the conclusion that mixture P-II (PETN with PA) and mixture P-III (PETN with HTPB) is actually incompatible.

Table 9. Results of the evolved gas volume for AP and polymers by method of vacuum stability test

Samples	The evolved gas volume, cm^3			V_R , cm^3
	M	E	S	
A-I	0.745			0.120
AP		0.072		
Viton A			0.793	
A-II	0.728			0.090
AP		0.072		
PA 12			0.746	
A-III	0.287			0.105
AP		0.072		
HTPB			0.320	

The results in Table 9 show that AP is compatible with all polymers and that is in accordance with the conclusion of values of difference in peak temperatures obtained from DSC analysis.

Conclusion

The compatibility of AP, HMX and PETN with Viton A, PA12 and HTPB was studied using the thermal method (differential scanning calorimetry) and vacuum stability test.

According to STANAG standard, mixtures with HMX and all polymer materials are considered as compatible by using both methods. Positive compatibility assessment for mixture of AP with all tested polymer materials is also confirmed by both methods.

However, only examined mixture of PETN with Viton A gives approval of compatibility by using the DSC and VST methods. Mixtures with PETN and other two polymer materials (PA and HTPB) by using the DSC method are considered as "moderately" incompatible which indicates that other method should be included for determination of compatibility. After analyzing the obtained results from VST method these two mixtures have shown and confirmed that system is incompatible.

According to these results it can be concluded that less than two methods cannot be used for sure for giving the opinions about compatibility of explosive materials and polymer materials.

Nevertheless, thermal method and vacuum stability test have their advantages and disadvantages. The greatest advantage of vacuum stability test in relation to the DSC is in the sample quantity. While for DSC analysis used mass of samples is approximately 1 mg of each studied materials, the

vacuum stability test is accomplished with 2.5 g of each material, which increases the possibility of physical contact between them. However, the vacuum stability presents some disadvantages, mainly due to composing the entire vacuum system before measurement, reaching the adequate vacuum at the beginning of examination and exposing to the highly poisonous product. Accordingly, all the procedures should be done correctly.

To sum up, results shown in this paper only testify the fact that the chemical compatibility is the complex parameter. Moreover, for better understanding the chemical compatibility, it is necessary to conduct several different methods based on different principle as confirmations of the given opinion on the results. Besides, the interpretation of the results should be done carefully and precisely.

Acknowledgement

The authors thank to the Ministry of Education, Science and Technological Development of Republic of Serbia for the support of the research through Contract No. 451-03-68/2020-14/200325.

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Received: 10.02.2021.
Accepted: 17.03.2021.

Difrencijalno skenirajuća kalorimetrija i test vakuum stabilnosti kao metode za određivanje kompatibilnosti eksploziva

Tokom proizvodnje, skladištenja i upotrebe eksplozivnih materija izuzetno je važno uočiti i pravovremeno sprečiti potencijalni rizik. Jedna od takvih potencijalnih opasnosti može biti izazvana prilikom kontakta eksplozivnih materija sa neeksplozivnim materijalima. Iz tog razloga različite analitičke i instrumentalne metode su razvijane i implementirane u okviru studije koja se bavi hemijskom kompatibilnosti eksploziva sa drugim materijalima.

U ovom radu su prikazani rezultati ispitivanja kompatibilnosti korišćenjem dve metode: termalna metoda-difrencijalna skenirajuća kalorimetrija (DSC) i test vakuum stabilnosti (TVS). Ispitivane su eksplozivne materije koje su često u upotrebi: Oktogen (HMX), Pentrit (PETN) i Amonijum perhlorat (AP) sa polimernim materijalima: Poliamid 12 (PA 12), Hidroksiterminirani polibutadijen (HTPB), Fluoroelastomer (Viton A). Provera kompatibilnosti je vršena korišćenjem kriterijuma kompatibilnosti definisanog u standardu STANAG 4147.

Ključne reči: energetski materijali, eksplozivi, polimeri, kompatibilnost, difrencijalno skenirajuća kalorimetrija, test vakuum stabilnosti.