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Capabilities of WITU relating to identification of explosives and the components containing thereof

Summary

The Military Institute of Armament Technology (WITU) is an accredited body performing expertises for prosecutors and courts, both civilian and military. Modern equipment allows a thorough analysis of the individual cases regarding the possession of explosives and ammunition within the meaning of law. WITU is equipped in such devices as DSC-TG, HPLC-PDA and GC-FID, used for confirming the origin of materials sampled from evidence. In addition, WITU owns an x-ray radiography system enabling preliminary, non-destructive testing, aimed at determining the structure of a compound and confirming its effectiveness.

Keywords expertise, DSC-TG, HPLC, GC-FID, x-ray image

Introduction

The Military Institute of Armament Technology (WITU) is an accredited body that handles ammunition analyses and implementations of modern solutions with regard to warfare agents. The Institute offers a broad range of analyses to the uniformed services as well as explosives (warfare agents) manufacturing companies. The studies carried out by the Institute are performed in compliance with its own validated methods, Polish standardization documents, as well as NATO standards. For many years, the activity of the Institute has focused primarily on extending ammunition resources, and examining ammunition components for their effectiveness.

Over many years of work, the staff of the Institute have acquired specialist expertise and experience in the evaluation of various types of munitions. This allowed the Institute to become an accredited body in the field of weaponry and ammunition. The modern equipment allows for fast, accurate identification of substances and the performance of reliable expertises for civilian and military courts and prosecutors.

Research facilities

The Explosives Research Department (ZBMW) has at its disposal extensive instrumental resources established to meet customer demand. The modern equipment is necessary for a number of analyzes confirming the origin of explosives. The department receives multiple inquiries as to whether the elements submitted for evaluation are considered ammunition within the meaning of the Arms and Ammunition Act [1], and whether their usage may pose a threat to life and health. Our staff members are able to address these inquiries by carrying out a series of necessary tests, including:

- Physical-chemical analyses identifying and determining the content and stability of compounds.
- External tests (X-ray radiography) enabling the assessment of the efficiency of ammunition components.

In order to perform physical-chemical analyses identifying substances as well as the extent of their degradation, a high-performance liquid chromatography (HPLC) system equipped with a PDA photodiode array detector is used. This device allows for qualitative and quantitative determination of the tested compound, based on not only on the retention time, but also on wavelength characteristics of a particular substance. Rapid identification of substances is possible, owing to the unique reference standards database managed by the Institute, including the majority of the most powerful blasting explosives, from the most common, such as trinitrotoluene (TNT), hexogen (RDX), octogen

(HMX), pentrite (PENT) etc. to the most sophisticated ones like HNIW (CL-20), FOX, NTO and many others. Very often, submitted as evidence are warfare agents containing propelling charge, such as cartridges in various calibers. In such cases, it is possible to determine the type of gunpowder and the stage of its degradation by using HPLC-PDA technique [2].

The identity of the material tested needs to be reconfirmed by using other analytical techniques. To this end, ZBMW uses the following devices: differential scanning calorimeter (DSC), thermogravimeter (TG) and calorimeter. These instruments enable the analysis of blasting, priming and propellant explosives [3], without the need for pretreatment (dissolving, sample concentration, etc.) [3,4].

Before disassembly of a munition component of unknown origin, X-ray radiography is performed, which is a non-destructive method of screening of ammunition or the components thereof. The information provided in this way determines whether or not the given component is fully functional and whether it can be safely disassembled in order to perform sampling.

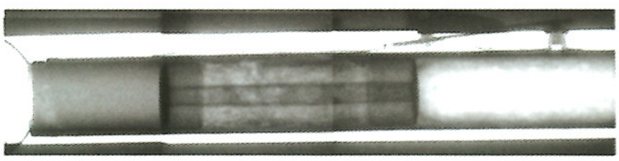
This article presents some exemplary forensic expertises in which the Institute has been involved.

Selected examples of forensic expertises

One of the examples of forensic expertises performed by the Explosives Research Department was the analysis of samples collected from grenade launcher submitted for testing (Fig. 1). Grenade launchers comprise certain components (e.g. primers) that can potentially contain explosives. The first stage of the analysis included non-destructive radiography tests, in order to obtain information about the presence of foreign matter in the tubes. The analysis of radiograms showed that the front sections of the tubes contained foreign objects in a form of narrow rods. Figures 2a-b show radiograms of launchers submitted for testing.

Fig. 1. Grenade launcher submitted for testing.

a)



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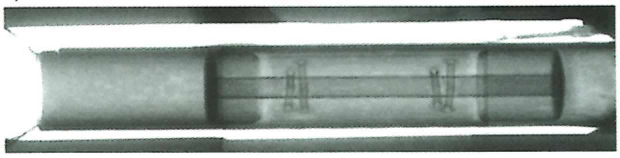
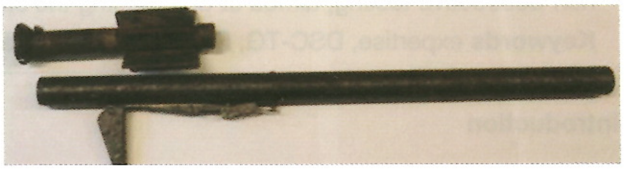


Fig. 2. Radiograms of the X-rayed grenade launchers.

The next step involved disassembly of launchers, which confirmed the conformance of the equipment with construction drawings and requirements, as regards the presence of the wooden shafts inside the tubes, used to stabilize the launcher's head (Figs. 3a-d). In one of the tubes, the shaft contained resilient, soft metal sheets, capable of unfolding after the launch (Figs. 3b,d). However, there were no traces of explosives found in any of the tubes.

a)

b)



c)

d)

Fig. 3. Launcher components after disassembly: (a),(c) – grenade launcher no. 1, (b),(d) – grenade launcher no. 2.

The disassembled launcher primers (Fig. 4) were subject to sampling in order to identify materials contained therein. The performed thermal analysis using a differential scanning calorimeter (DSC) led to the conclusion that the primer of grenade launcher no. 2 contained residual amounts (traces) of lead trinitroresorcinate (priming explosive), as indicated by an exothermic peak corresponding to the decomposition of a sample, with a maximum at 305.9ºC (Chart 1).

a)

b)

Fig. 4. Primers in the analyzed grenade launchers no. 1 (a) and no. 2 (b).

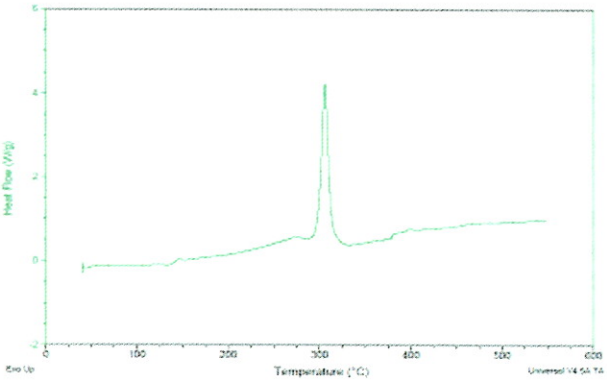


Chart 1. The result of thermal analysis of the material sampled from the primer of grenade launcher no. 2

Table 1 The values of characteristic temperatures and the heat of physical-chemical transformation of the material (lead trinitroresorcinate) sampled from the primer, obtained using scanning microcalorimetry DSC

Item number	Parameter	Primer material
1.	Peak Temperature [ºC]	305.9
2.	Onset Temperature [ºC]	298.5
3.	Heat of transformation [J/g]	210.3

The heat of degradation obtained with this method only gave an indication of trace quantities of undegraded lead trinitroresorcinate, as shown in Table 1. Under such conditions, an explosive did not exhibit priming characteristics and did not pose a danger to third parties. Therefore, the ammunition component was not hazardous, but rather of historical nature. In summary, neither the grenade launcher nor the material sampled from the primer interior posed a danger and they were not considered explosives within the meaning of the Arms and Ammunition Act [1].

Another example of expertise performed by the ZBMW staff was the testing of the properties of gunpowder samples with regard to safety and disposal possibilities. To assess the technical condition of the samples, a visual inspection of gunpowders and their packagings was performed. The next step was to determine the chemical stability of gunpowder samples and to conduct thermal analysis with the use of differential scanning calorimetry.

Visual inspection of the containers with gunpowders revealed the presence of the various types of infiltrations and deposits, signs of corrosion on metal parts of packagings as well as inconclusive labeling of containers (Fig. 5a). Gunpowder grains were agglomerated, discolored, with clear signs of mechanical damage (Fig. 5b).

In order to assess the changes within the samples received, thermal analysis was performed using a differential scanning calorimeter. The analysis confirmed that the gunpowders in the containers were in a state of degradation. Figure 2 presents the DSC curve of an exemplary gunpowder sample tested, showed in juxtaposition with a nitrocellulose standard for better illustration of sample degradation. Table 2 shows the characteristic temperatures and the physical-chemical transformation heat values of the tested gunpowder and nitrocellulose standard.

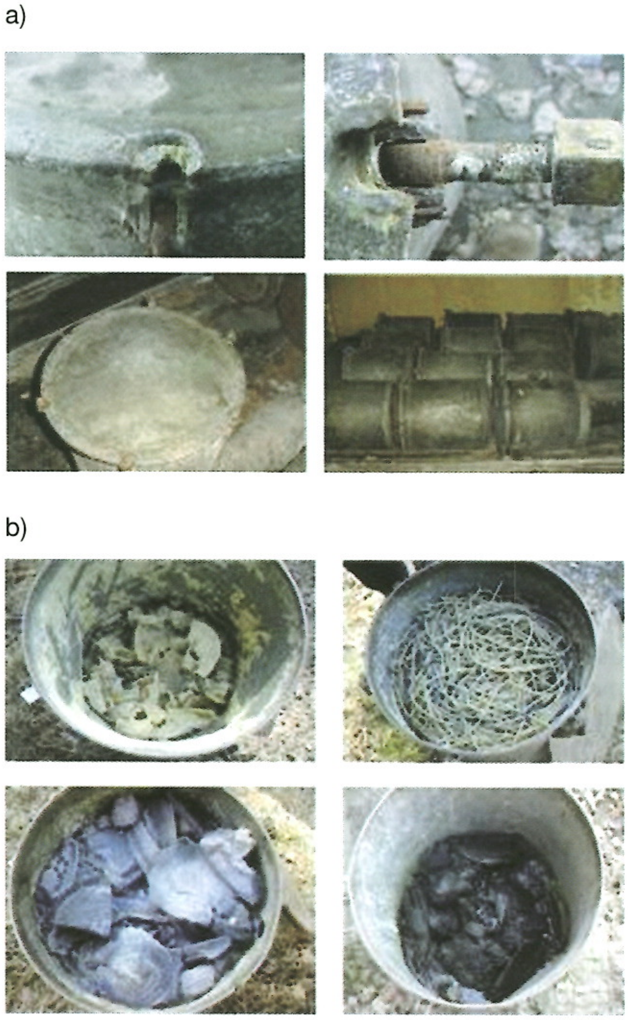


Fig. 5. Condition of the containers and gunpowders submitted for testing.

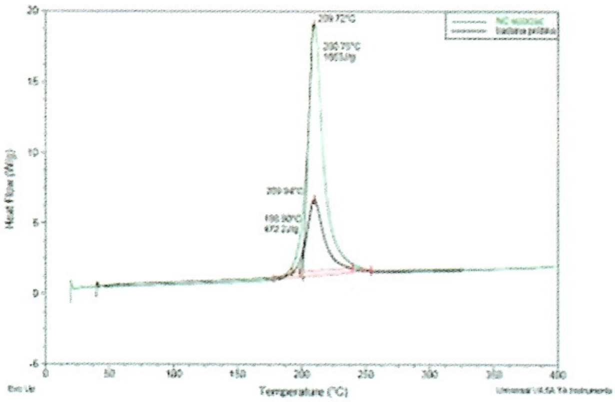


Chart 2. Results of thermal analysis of a nitrocellulose standard and an exemplary gunpowder sample being the subject of expertise.

Table 2 Characteristic temperatures and the physical-chemical transformation heat values of an exemplary gunpowder sample submitted for DSC analysis

Item number	Parameter	Nitrocellulose standard	Tested gunpowder
1.	Peak Temperature [ºC]	209.7	209.9
2.	Onset Temperature [ºC]	200.8	198.9
3.	Heat of transformation [J/g]	1663.0	472.2

The performed tests showed that the transformation heat of the gunpowder sample had a much lower value as compared with a nitrocellulose standard, which indicated a significant decomposition of the tested gunpowder. The condition of the gunpowder allowed for its transport, albeit with particular caution. Hence, due to the potential danger, the utilization of the materials had to be supervised, so as to comply with all necessary precautions.

The last example concerns an expertise on the ammunition components delivered to WITU. The material evidence in a form of cartridges were disassembled in order to collect samples for analysis. The disassembled cartridges were in poor technical condition, as shown on Fig. 6 and 7.

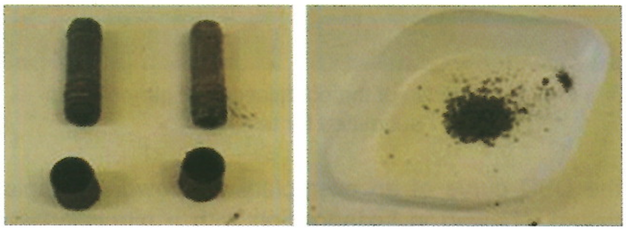


Fig. 6. Sample taken from material evidence no. 1.

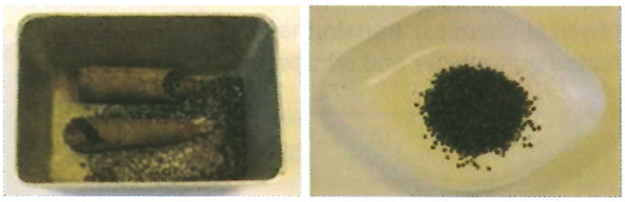


Fig. 7. Sample taken from material evidence no. 2.

Fig. 8. The cases of disassembled cartridges after cleaning.

After disassembly of the cartridges, it was found that they likely contained nitrocellulose gunpowder. After cleaning cartridge cases with 0.1% HCl, it was possible to read the year of manufacture. The majority of the cartridges were manufactured in the late 1940s (Fig. 8).

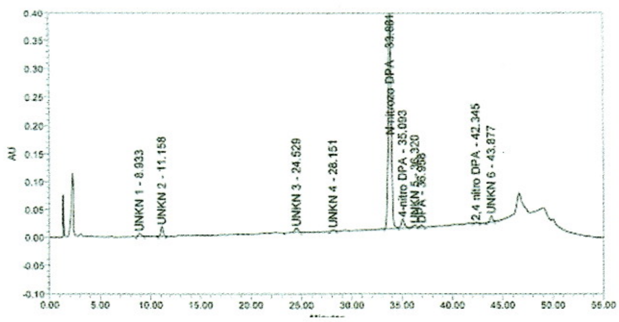
A preliminary analysis of the sampled material carried out using the calorimetric method confirmed that it was a propellant. Table 3 presents the heat of combustion values and combustion rates determined for the sampled material, compared with a standard reference material – nitrocellulose gunpowder.

Table 3 Combustion rates and the heat of combustion values of the tested materials

Evidence no.	Combustion rate [s]	Heat of combustion [J/g]	Combustion rate [s] of gunpowder standard NC	Heat of combustion [J/g] of gunpowder standard NC
1	0.8717	3401	1.0584	4178
2	0.8676	3417		

The sample analysis revealed high combustion rates and high calorimetric parameters, similar to those of the standard nitrocellulose gunpowder. The tests confirmed, that the sampled material was a propellant – nitrocellulose gunpowder. A slightly lower heat of combustion value and higher combustion rate (as compared with the reference) may indicate degradation of the sampled material.

a)



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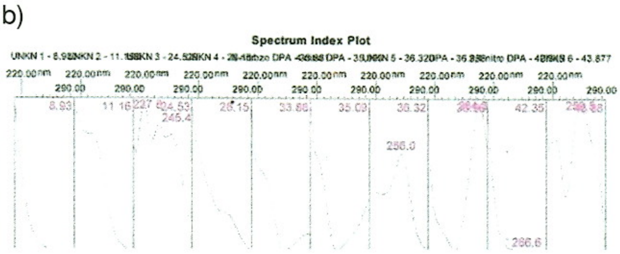

Chart 3. a) An exemplary chromatogram of nitrocellulose gunpowder sampled from material evidence no. 1, containing a diphenylamine stabilizer in a state of almost complete degradation, (b) UV spectra of identified and unidentified degradation products of the main diphenylamine stabilizer.

In order to fully identify and assess the aging progress, a chromatography test was performed based on STANAG 4620 [5]. Shown below is an exemplary chromatograph of the sample taken from material evidence no. 1 (Chart 3).

The obtained chromatograms and UV spectra were compared against the standards within a database and it was found that peaks visible in the chromatogram (sample taken from material evidence no 1) correspond to diphenylamine and its degradation products: N-nitrosodiphenylamine, 2,4-dinitrodiphenylamine, 2-nitrodiphenylamine and 4-nitrodiphenylamine. Subsequently, based on chromatographic data, the contents of individual components were calculated. Table 4 shows the test results of samples taken from material evidence no. 1 and 2. The analyses confirmed the presence of a diphenylamine stabilizer, which is often used in this type of nitrocellulose gunpowder, certain degradation products thereof, as well as the lack of other additives.

Table 4 Contents of stabilizer and its degradation products in material evidence no. 1 and 2, calculated based on chromatographic data

Evidence no.	Stabilizer [%]	Stabilizer degradation products [%]		
	DPA	N-nitroso DPA	2-nitro-DPA	4-nitro-DPA
1	0.01	0.91	0.08	0.06
2	0.13	0.42	0.04	0.09

In conclusion, HPLC and calorimetry-based analyses have confirmed that the tested cartridges contained nitrocellulose gunpowder in a state of degradation, as evidenced by high N-nitrosodiphenylamine levels, and the appearance of other degradation products: 2- and 4-nitrodiphenylamine, while only a negligible amount of diphenylamine stabilizer was present. Generally, the level of the main stabilizer in gunpowder must not fall below 0.2%, as lower levels may lead to uncontrolled reactions, such as self-ignition. Thus, the levels detected in the tested gunpowders render them unstable and dangerous. The owner of such material could (even unknowingly) create a threat to one's health or even life.

Conclusions

Use of modern analytical techniques quickly provides information on the significance of ammunition components submitted for testing, pursuant to the Arms and Ammunition Act.

A preliminary radiography analysis allows for screening of the tested warfare agent or its components without the need to disassemble or unscrew thereof. It is extremely important to conduct an evaluation of the condition and contents of a given warfare agent or its element, which affects further decisions as to the rationale and feasibility of sample collection for chemical analyses. The radiographic test also reveals details of construction of a particular warfare agent, especially in the cases of expertises concerning old ammunition or ammunition lacking technical documentation.

Thermal analysis is used to obtain information whether the test sample contains an explosive material. HPLC analysis facilitates precise identification of the sample as well as qualitative and quantitative determination of the compounds contained therein.

The examples of expertises presented in this article confirm WITU's capabilities with regard to the identification of explosives or the elements containing thereof. Thanks to continuous teamwork of the Explosives Research Department staff, their knowledge and experience, opinions and answers to legal inquiries are prepared in a professional manner. Analytical equipment facilitates adequate and rapid identification of explosives and objects containing thereof. Complete and professional expertise can only be performed on samples submitted for testing (even in milligram quantities) by the contracting authority.

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Translation Rafał Wierzchosławski