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Research paper

Persistence of TNT Traces in the Presence of Compounds from Battery Cells

Wojciech Pawłowski¹, Monika Karpińska^{*,2},
Katarzyna Razarenkow-Pawłowska³

¹ *Department of High-Energetic Materials, Faculty of Chemistry, Warsaw University of Technology, 3 Noakowskiego Street, 00-664 Warsaw, Poland*

² *Kielanowski Institute of Animal Physiology and Nutrition of Polish Academy of Science, 3 Instytutcka Street, 05-110 Jabłonna, Poland*

³ *Chemistry Department, Central Forensic Laboratory of the Police, 7 Ujazdowskie Avenue, 00-583 Warsaw, Poland*

* *E-mail: mkarpinska@ch.pw.edu.pl*

Abstract: Nowadays, laboratory forensic examinations are based on elaborated and verified protocols, allowing for quite precise identification of the type of explosive used in post blast residues. Non-degraded explosives persist in trace quantities mainly on the broken components of explosive devices. Due to this, special care must be taken in order to prevent destruction of minute quantities of explosive material during examination. Apparently quite a big problem is caused by components of electrical cells being the source of reactive chemical compounds that affect the level of identified traces of explosives.

The present paper describes the influence of substances present in alkaline batteries cells and Leclanché cells on the decomposition of trace amounts of TNT. Reaction of TNT with battery components was conducted in solution at room as well as the temperature of boiling methanol. Quantitative analysis of the decrease in TNT levels in methanol extracts was carried out by means of GC/MS.

Keywords: forensic science, explosives, TNT, GC-MS, trace analysis, battery cells

Supporting Information (SI) is available at:

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1 Introduction

The threat of terrorist attacks using explosives is constantly at a high level. The use of explosive devices in bomb attacks, *e.g.* in Iraq and Afghanistan, has shown an increased interest in homemade explosive devices. Criminals obtain explosives from various sources, *e.g.* by smuggling, theft, collection, recovery of materials remaining in post war conflict areas. The relatively easy access to explosives' precursors, the simplicity of construction of homemade explosive devices and the wide access to information (*e.g.* through the Internet) regarding the construction of so-called homemade explosive devices constitute increasing threat factors. Electrical methods for initiating explosives have gained particularly high popularity in terms of their use in the construction of bombs employed by terrorists. Therefore, most explosive devices are presently constructed on the basis of electronic components, which are powered by an appropriate source of electricity, usually regular batteries. These cells are made up of active chemical components, which become dispersed in large quantities around the epicenter after a bomb explosion. Several types of electrical cells with chemical components can be distinguished, namely: alkaline cells (KOH, ZnO, MnO₂), zinc-carbon Leclanché cells (MnO₂, NH₄Cl, ZnCl₂, Zn, C), nickel-cadmium cells (Ni, Cd) and others [1]. Alkaline batteries (Figure 1(a)) and Leclanché zinc-carbon cells (Figure 1(b)) are the most commonly available types.

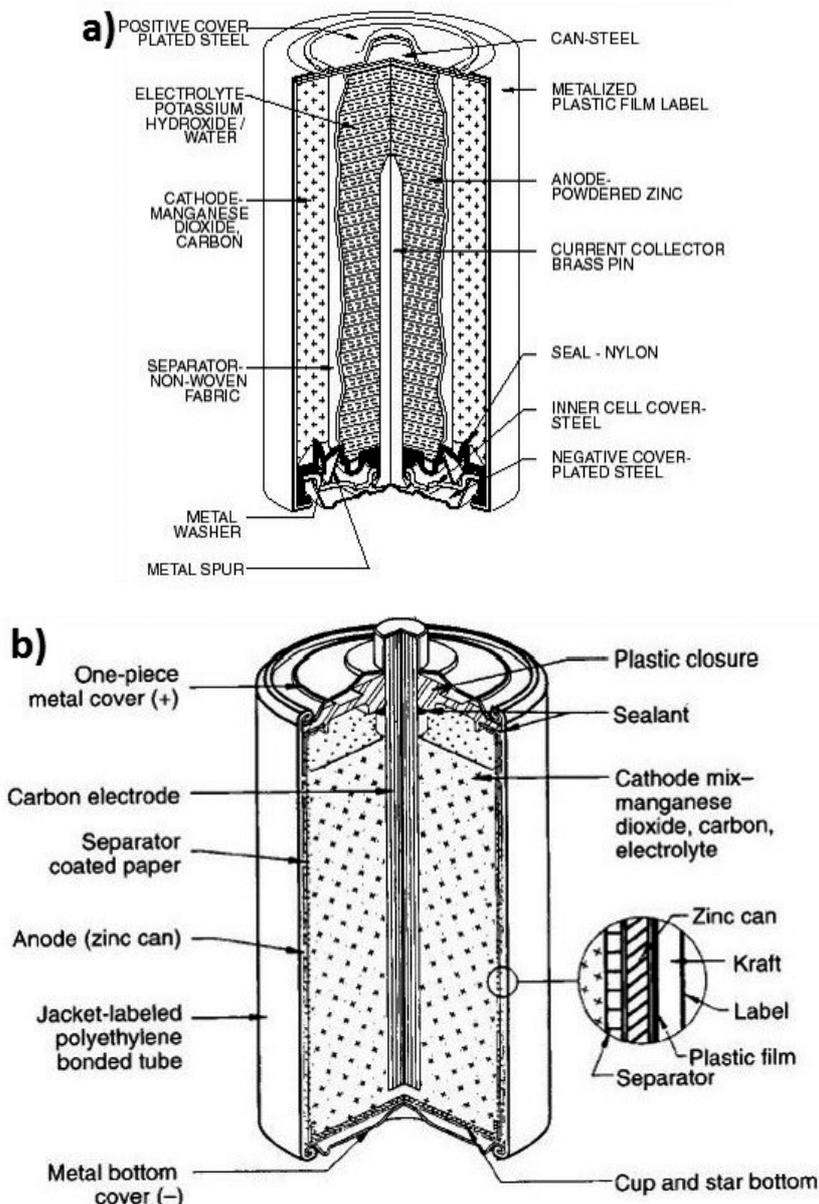


Figure 1. Cross-section of (a) an alkaline battery, and (b) a Leclanché cell

Dispersed battery components (Figure 2), as chemically reactive substances, may interact with other chemical compounds *i.e.* post blast

residues, and have a significant effect on the chances for the recovery of trace amounts of explosives for forensic examination. This may produce a situation where subsequent identification of the explosives used would become practically impossible. The most common methods utilized for the recovery of post-blast trace explosives include extraction with methanol, ethanol, acetone, or water [2]. As a result of this process, concentrated solutions are obtained, which contain, in addition to trace amount of nitro-compounds, an entire range of chemical compounds constituting the so-called matrix. As the explosive is usually found on broken explosive parts, in amounts which do not exceed milligram quantities, it is the matrix which poses identification problems. The matrix may contain reactive chemical compounds which hinder the direct identification of the explosive used. In order to eliminate any matrix effects, various extract purification processes can be used, *e.g.* by means of SPE [3] or SPME [4] methods. Regrettably, even the most sophisticated methods for matrix separation will not be effective if, during the extraction, the explosive being identified comes into reaction with reactive matrix compounds, becomes degraded or converted into other chemical compounds, before it has been separated. Its trace concentration in the extract may fall below the limit of detection, and hence cannot be detected. With this in mind, the influence of the chemical substances found in electrical batteries on the persistence of traces of explosives, identified in forensic laboratories, should be examined.

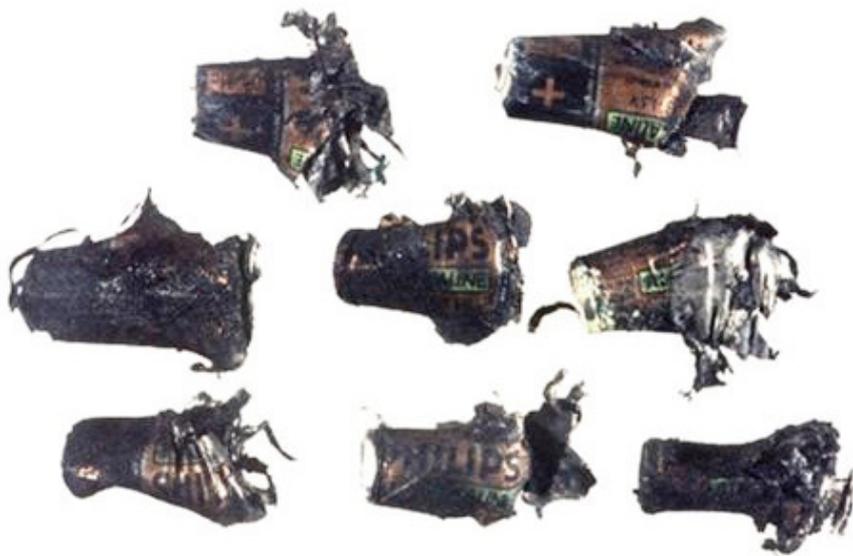


Figure 2. Electrical alkaline batteries damaged as a result of an explosion

Considering that the active material in a battery is enclosed in a metal housing, and in the construction of the explosive charge the battery is usually at a distance from the explosive materials, it implies that the interaction of the active substances of the battery and the explosive material is not direct. It has been assumed that since there are large or small fragments of the battery housing at the place of the crime incident (Figure 2), they also contain some quantities of active substances from the battery, with the original chemical composition of the battery intact. We considered a state in which an explosive charge breaks the battery and disperses the active products of the battery filling. The extreme situation in which the battery does not break is not a problem, because then the active substances from the battery will not contaminate the environment. It was assumed, that the explosive transformation is fast enough that its thermal impact and explosion products have no significant effect on changing the chemical composition of an active battery filling.

According to literature reports and from the perspective of chemical properties, a trinitrotoluene (TNT) particle reacts easily with nucleophiles, which may be the hydroxide anion (OH^-) originating from potassium hydroxide (KOH) in an aqueous environment, as well as in an alcoholic environment [5]. Hantzsch and Kissel [6] treated TNT with potassium alkoxide and obtained an addition product, the so-called Meisenheimer complex. Chemically, this complex demonstrates ion (salt) properties. Studies have shown that mineral acids (such as sulphuric acid) contribute to the decomposition of the salt, which is accompanied by release of nitrogen oxides. At the same time, a product of further conversion of TNT, which includes methyl group oxidation ($-\text{CH}_3$), is precipitated. Other authors [7] concluded that in the presence of an excess of potassium hydroxide, a Janovsky complex is formed. When applying an excess of potassium hydroxide to TNT, no formation of such complexes was observed, but instead coloured Meisenheimer complexes were formed. Complexes of structures similar to Meisenheimer complexes are also formed as a result of TNT and other aromatic polynitrocompounds reacting with phenolate anion, formed from ketones (*e.g.* acetone) under the influence of hydroxide ion [8]. The reactions in question, due to the intense colour of the compounds being formed, have often been utilized in colour tests for preliminary identification of TNT, as well as other explosives in the aromatic polynitrocompound group [9, 10].

Attention should be also paid to the reactivity of borosilicate glassware (3.3, SIMAX: SiO_2 80.6%, B_2O_3 13%, Al_2O_3 2.4%, $\text{Na}_2\text{O} + \text{K}_2\text{O}$ 4% types). It is characterized by high chemical resistance with complex processes [11-17]. It also exhibits reactivity with OH^- groups.

Laboratory glassware can be replaced with polypropylene (PP) vessels, however PP is not as resistant to temperature variations as borosilicate glassware and therefore is used in analyses conducted mainly at room temperature.

Abdul-Karim *et al.* [18] outlined how the traces of explosives after detonation decompose, and they drew attention to the possible impact of the environment on the results of analyzes aimed at revealing the type of explosive used. From our many years of practice in disclosing elements of explosive devices after detonation (especially TNT charges) at the scene of an event and analyzing trace residues for their quantities, it appears that identification by qualitative analysis and gas chromatography is often more difficult and sometimes even impossible when the explosion is electrically initiated by means of a device powered by electrical cells. Therefore, the necessary comparative tests have been carried out. Under the same ambient conditions, 0.5 kg TNT charges were fired, except that in one case the initiation was carried out with a fuse and in the other experiments by a delayed device with Leclanché and alkaline batteries placed directly next to the TNT charge. It was found that the results obtained differ significantly. In the case of chemical analysis of traces taken from the residue after firing the TNT charge with a fuse, the identification of TNT was consistently positive. However, in experiments in which the initiating devices were powered by electrical cells, some problems were encountered during the chemical identification of trace amounts of TNT.

The observations described above encouraged the authors of the present paper to determine the influence of reactive substances in battery cells on the persistence of trace amounts of TNT found in post-blast residues. The chemical interaction between TNT and the reactive substances from the cells of the most popular batteries, in a protic solvent such as methanol, was examined. Methanol is also a solvent recommended by ENFSI [2], because it seems to be fairly unreactive. This allowed verification of the general protocols for preparation of extracts for chemical identification, collected from an explosion epicenter and scattered fragments of an explosive device. TNT was selected due to its widespread use in military explosives and devices, as well as its present application in arms and civil industry. In addition, the conclusions drawn on the basis of the TNT studies may be applied to all explosives based on the polynitroaromatic group, because they undergo the same types of side reactions. In order to find out how TNT behaves in glassware, the samples with no alkaline agents were also prepared. As the process of extraction is carried out in boiling solvent, as one method which yields better results, the analyses were initially conducted in boiling methanol, which were then followed by analyses at room temperature, for comparison purposes. Quantitative analysis

was conducted by means of gas chromatography with mass spectrometric detection (GC/MS). The method is widely used in the analysis of trace explosives, being one of the most popular methods used, and is recommended by ENFSI. It allows traces at the ppb level to be detected and is the most suitable method for the analysis of post blast residues [2, 19].

The intention of the authors of this publication was to conduct laboratory work with planned experiments under field conditions in mind, using TNT and alkaline batteries and Leclanché cells. After collecting the traces and carrying out the tests in accordance with the procedure developed in this article, it was found that the results corresponded very well with the results obtained in the model studies in the laboratory.

2 Materials and Methods

2.1 Reagents and materials

The following solvents were used as reagents: methanol (p.a.) (POCh, in Polish: Polskie Odczynniki Chemiczne), chloroform (p.a.) POCh. Batteries: Duracell Ultra M3 Technology AA LR6-MN1500 1.5V (alkaline), Ansmann Special R20 1.5V. The explosive used in the examinations was trinitrotoluene (TNT) in the form of flakes „Nysa”, produced by ZTS Nitron S.A. (Bydgoszcz, Poland). The analyses were conducted in borosilicate glassware, SIMAX.

2.2 Sample preparation and analysis

The analysis of the decomposition of TNT at room temperature (21 °C) and at the boiling point of methanol (64.7 °C) was conducted in the presence of an active mass collected from a battery, and also for a system using only pure solvent. The active mass of the battery was prepared by disassembly of the tin casing, removing the electrode and crumbling the remaining part. The weighed active mass (15.00 g) was transferred to a 500 mL glass beaker containing methanol (250 mL). The resulting mixture was divided into two parts; one was kept at ambient temperature, whereas the other part was heated to the boiling point of methanol. Subsequently, a TNT sample (0.1977 g) was added each flask. At relevant time intervals, portion of approximately 2 mL solution were removed by syringe and transferred to a Petri dish. They were immediately (in no more than 30 s) subjected to evaporation until dry by means of forced air flow. 2 mL of an internal standard (0.1 wt.% 1,4-dinitrobenzene, *p*-DNB, solution in chloroform) was added by pipette to the dried sample on the Petri dish.

Solutions prepared in this manner were quantitatively transferred to vials and analyzed by chromatography.

2.3 Construction of calibration curves

Calibration curves were plotted based on a series of measurements run on GC/MS instrumentation. A calibration curve is a plot demonstrating the relationship between the peak area for known analyte concentrations *vs.* peak area of a fixed concentration of a standard. *p*-DNB was selected as the standard, being a substance which fulfills all of the analytical criteria for the purposes of these experiments. The method consists in the preparation of standard and analyte solutions of the same concentration (0.1%). Whereas the amount of internal standard is always the same (usually the same volume of standard is added), various volumes of analyte are added (of the same concentration as the standard).

2.4 Methods

Prepared samples, in the form of extracts, were analyzed using a GC789A gas chromatograph coupled with a mass spectrometer VILMSD 5975C (quadrupol) by Agilent Technologies. The chromatograph was equipped with an HP-5MS column, 15 m length, 0.25 mm diameter and 0.25 μ m film thickness. The analyses were conducted under isothermal conditions (120 °C). Other parameters were:

- split 1:40,
- gas (He) flow rate: 1.5 mL/min,
- injection volume: 1 μ L,
- scanning range m/z = 29-550,
- analysis time 13 min.

Before the analysis, a calibration curve in the concentration range 0.005-0.5 wt.% TNT was generated using *p*-DNB as the internal standard. Calculations were performed basing on the TNT peak areas for ion m/z = 210 as well as *p*-DNB peak areas for ion m/z = 168. A linear relationship was obtained in the concentration range 0.005-0.02 wt.% TNT.

During the experiments, the following assumptions for the extraction process were adopted in order to make the results comparable:

- In each experiment, the initial concentration of TNT in methanol was the same and amounted to 0.1 wt.%.
- Analysis was conducted over 60 min; 5 samples were collected within first 10 min and 5 samples within the remaining 50 min.

The calibration curve of TNT concentration *vs.* ratio of peak areas of analyte

and standard (TNT/*p*-DNB) served to give the variations in TNT concentration vs. time, and the relationships obtained were presented as plots.

3 Results and Discussion

Extraction with concentration can be conducted (as required) by using two methods: “fast” and “slow”. The “slow” method is based on washing the material with methanol, while the extract is left for further concentration under ambient conditions. Such a process lasts for several dozen hours. The “fast” method is based on washing the material with methanol, while the extract undergoes concentration at boiling temperature. Such a process lasts up to several hours. It was evident that chemical reaction at a higher temperature is faster (a temperature increase of 10 K affords 2-4 times higher speed of reaction – the van’t Hoff law).

As a result, in presence of an aggressive matrix at the solvent’s boiling point, the analyte can react far quicker with the matrix compounds. This prompted us to examine how trace amounts of TNT would behave in contact with matrix compounds derived from alkaline batteries and Leclanché cells during methanol extraction. We started by conducting a zero trial, which means that the TNT methanol solution was concentrated using the “fast” and “slow” methods in laboratory glassware (Figure 3). It was observed that at both temperatures, the TNT concentration decreased, however the curves differed in their trajectories. The trajectory of the curve obtained by evaporating the solvent at room temperature resembled a straight line with a slight inclination, causing a concentration decrease of only 0.01% over a time of 60 min. In the “fast” trial, conducted in boiling methanol, a two-stage TNT concentration decrease was observed. At the first stage, lasting *ca.* 15 min, the process was faster, and then the curve became flat, while over 45 min it resembled a straight-line trajectory with a slight inclination. A summary drop in TNT concentration over the 60 min was higher than in the case of the “slow” method, amounting to 0.02%. The reason for this phenomenon is the temperature at which the process is conducted, which is in line with the van’t Hoff law. The observed decrease in TNT concentration in boiling methanol can be explained by a reaction similar to the reactions of aromatic polynitro compounds with methoxide anions in an alkaline environment mentioned earlier in [8]. In the formation of the methoxide anion, a significant role can be attributed to the alkaline properties of glass. At ambient temperature, the TNT concentration changes only slightly during the process. The cause of a concentration decrease could be the same

as the one proposed in the case of boiling methanol, although the influence of TNT adsorption on the walls of the glass vessel cannot be fully excluded here. In the case of the “fast” extraction in boiling methanol, a certain amount of TNT becomes decomposed. This effect should be taken into account when assessing the variations in TNT levels during the trials conducted in the presence of other reactive substances.

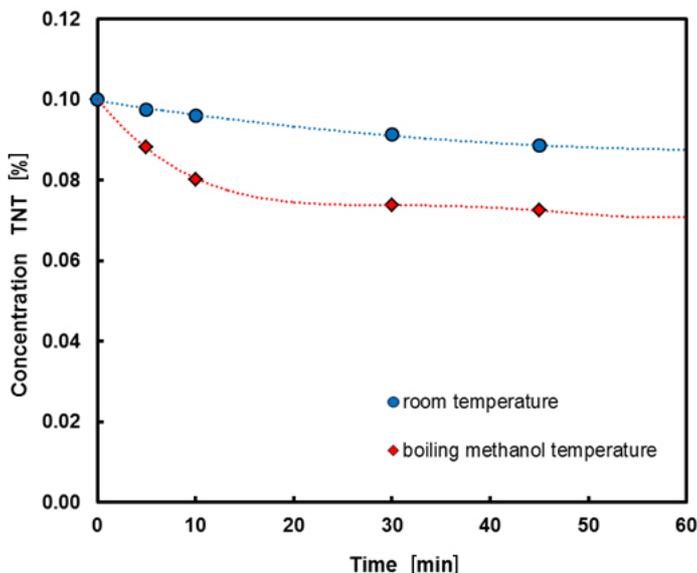


Figure 3. Change in TNT concentration at room temperature and at the temperature of boiling methanol (see SI, Table S1)

As the next step, “fast” extraction in boiling methanol for a matrix derived from an alkaline battery and a Leclanché cell was conducted, with the results shown in Figure 4. The shape of the curve obtained for alkaline batteries is quite similar to the curve derived from the reaction of TNT decomposition [20]. Here, the source of hydroxide ions (OH^-) is potassium hydroxide that had been adsorbed earlier onto the particles of the battery mass. The largest decrease in TNT concentration was observed within 10 min. Following this, a slow change in TNT concentration was observed over a longer extraction time, 10 to 30 min. From 30 min onwards, the TNT concentration remained practically unchanged. This happens because the KOH from the cell of an alkaline battery, dissolved in methanol, generates a nucleophilic reaction with the TNT and causes a sudden drop in TNT concentration in a short time,

then becomes asymptotically close to complete decomposition. In cases where the extraction was conducted in a system with no alkaline factor (Leclanché cell), it can be clearly observed that a quite sudden decrease in the TNT concentration in the extract also takes place within the first 3 min, however it is not as rapid as in the case of an alkaline battery, where it occurs as early as after 1 min at the same level (*ca.* 0.02%). Subsequently, after 6 min a concentration increase of about 0.01% mass was observed, followed by stabilization at a steady level, amounting to *ca.* 0.03% after approximately 10 min. The initial rapid decrease in TNT concentration could be caused by TNT adsorption on the cell's permanent components, such as carbon and MnO_2 . Under the influence of the solvent and heating at the boiling point of methanol, a process of fast partial desorption of TNT from the mass of active battery components takes place. This results in the increase in TNT concentration. The above mentioned carbon is a perfect adsorbent that could have an effect on the irreversible adsorption, by causing desorption to stop at the level of 0.03% TNT concentration.

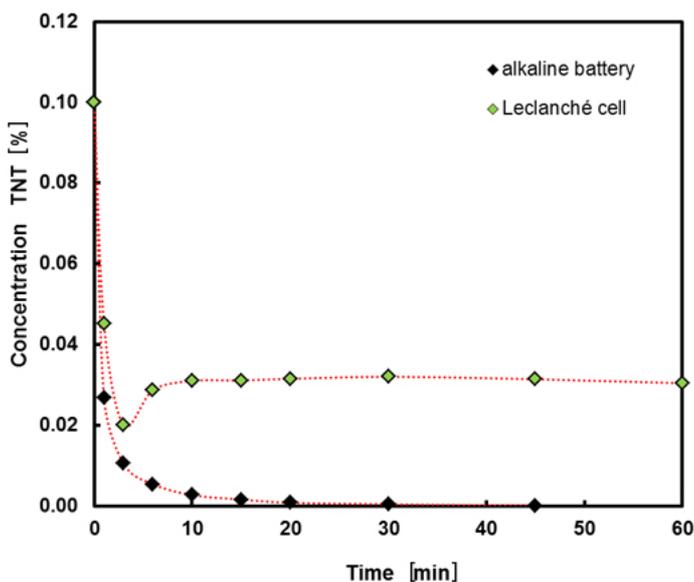


Figure 4. Change in TNT concentration as a function of extraction time, for a mass of active substances from an alkaline Duracell Ultra M3 battery and a Leclanché cell at the temperature of boiling methanol (see SI, Table S2)

This study was followed by “slow” extraction at room temperature. The trajectories of concentration decreases with time are presented in Figure 3. The curve of TNT concentration change for an alkaline battery initially behaves in a similar manner to that for a Leclanché battery. Additionally, it is possible here to observe the stages typical of adsorption-desorption processes, which are characterized by a quite rapid decrease in TNT concentration, followed by an increase. For extraction from the active substances from an alkaline cell, the adsorption takes place slightly faster up to 6 min, then the TNT concentration keeps increasing slightly until 15 min of extraction, and, after a short 10-min period of stabilization, it decreases systematically down to a level of 0.015% during 60 min. On the other hand, for extraction from the active substances from a Leclanché cell, desorption begins as late as after 10 min of the process, with a slight increase lasting for about 15 min of extraction, and becomes stabilized at *ca.* 0.035 wt.% TNT.

By comparing the TNT concentration changes at ambient temperature for matrixes produced from an alkaline battery and a Leclanché cell battery, it can be seen that the trajectory curves are not as markedly different as in the case of the “fast” extraction at the temperature of boiling methanol, although the TNT concentration after an hour of extraction is significantly different. This is connected with the influence of the KOH in alkaline cells, as described earlier.

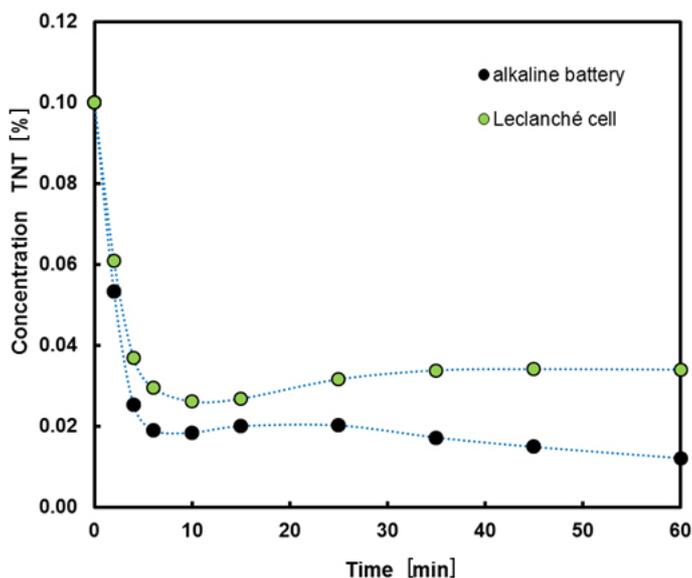


Figure 5. Change in TNT concentration as a function of extraction time for a mass of active substances from an alkaline Duracell Ultra M3 battery and a Leclanché cell at room temperature (see SI, Table S3)

In course of further examinations, being aware of the above findings, a solution for avoiding the loss of the analyte TNT during the extraction was sought. To this end, an attempt to neutralize an alkaline pH by adding hydrochloric acid solution to methanol before conducting an extraction was undertaken. The amount of hydrochloric acid added to the methanol was chosen in such a way as to obtain an extract during the extraction process with a mildly acidic pH, 4, when tested using a water-moistened pH paper. These attempts were conducted using the “fast” extraction method in boiling methanol and the “slow” extraction method at ambient temperature, similar to those methods described above. The results obtained are presented in Figure 6.

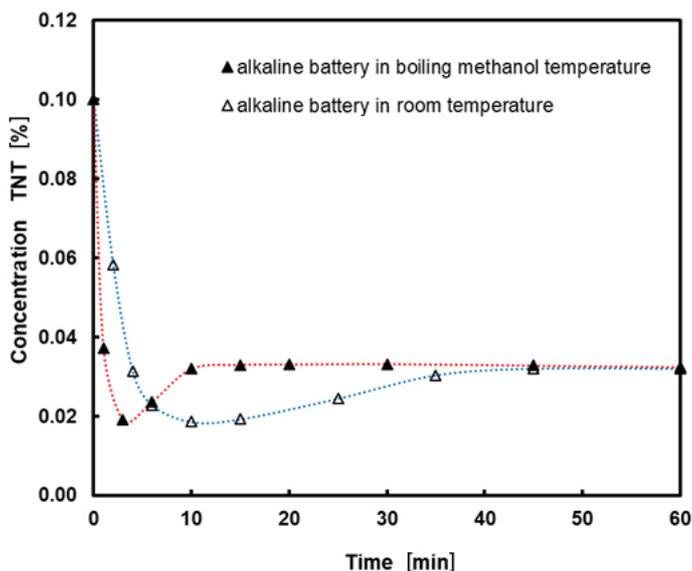


Figure 6. Change in TNT concentration as a function of extraction time in the “fast” and “slow” methods with the addition of hydrochloric acid, for a mass of active substances from a Duracell Ultra M3 alkaline battery (see SI, Table S4)

The combined results indicated that in both “fast” and “slow” extraction methods, after 45 min the TNT concentrations were at the same level, 0.035%, and remained unchanged thereafter. Any differences stem from the fact that in the case of the “fast” method, the TNT concentration becomes stabilized as early as 10 min of extraction. This can be explained by the fact that in these processes there are no reactions with KOH as it has been neutralized, so the sole influence on the TNT level in methanol solution is exerted by adsorption-

desorption processes, which occur faster at the temperature of boiling methanol.

When observing the changes in TNT concentrations during methanol extraction with the addition of HCl_{aq} at room temperature with a mass of active substances from an alkaline battery, it was concluded that they were similar to the course of the TNT concentration changes in extraction performed by the same method for a mass of active substances from a Leclanché cell (Figure 7).

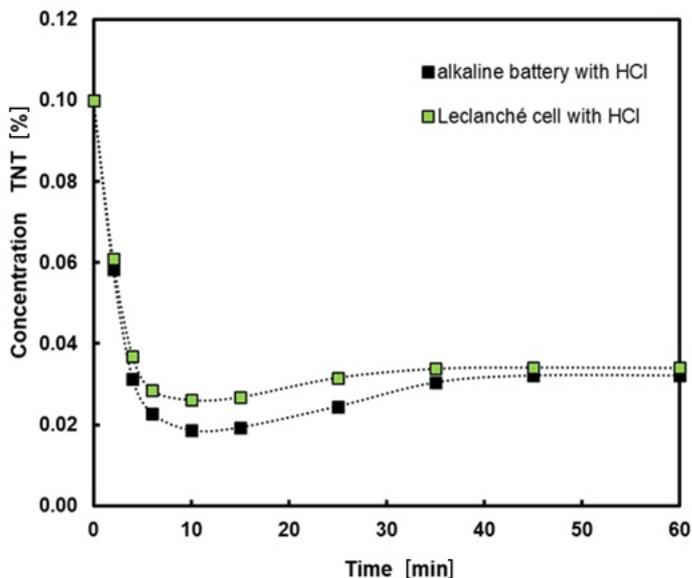


Figure 7. Change in TNT concentration as a function of extraction time at ambient temperature with addition of hydrochloric acid in methanol solution, for a mass of active substances from a Duracell Ultra M3 alkaline battery and a Leclanché cell (see SI, Table S5)

The differences between the curves representing changes in the TNT concentration stem from the fact that in the case of an extraction carried out in the presence of the mass from alkaline batteries, the adsorption-desorption processes are slightly deeper. The maximum decrease in TNT concentration amounted to 0.019% in 10 min, rising to a similar value during 60 min.

4 Conclusions

Universally utilized protocols for obtaining an extract from recovered evidence have been verified. We found that in the process of extraction of post-blast

residues containing TNT at the temperature of boiling methanol, the fastest decomposition of the explosive material is caused by reactive substances originating from exploded alkaline batteries, which may significantly affect the analytical result of the identified explosive.

It was found that in the case of extractions with batteries not containing alkaline agents (Leclanché cells), the contents of such batteries also cause loss of TNT residues, however the decrease in TNT concentration is much lower than as in the case of alkaline batteries.

Extractions conducted with a solution of hydrochloric acid in methanol, aiming to obtain a neutral or slightly acidic extract in the case of a matrix containing active substances from an alkaline battery, yielded results which were similar to those obtained with a matrix of substances contained in a Leclanché cell and leads to a smaller decrease in concentration of trace amounts of TNT, thus facilitating the identification of the explosive used.

The use of hydrochloric acid for neutralizing the analyzed matrix does not have any effect on the course of an extraction in the presence of Leclanché cell components, however it significantly improves the possibility of extraction of TNT in the presence of alkaline battery components.

The application of pH control of the material for extraction and ensuring a neutral pH of the methanol solution during extraction, provides the possibility for better detection of TNT residues by chromatographic methods.

The experiments conducted demonstrated the effect of alkaline substances contained in electrical cells on the possibilities for revealing and identifying trace amounts of TNT remaining after a detonation, which is a key issue in forensic practice.

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