AARMS

TECHNOLOGY

Some experience with trace analysis of post-explosion residues

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The paper deals with possible applications of GC-ECD and IC to specification of post-explosion residues of secondary explosives. Technical TNT and dynamite Danubit 2 have been used as real secondary explosives: at present they are the most frequently abused explosives on the territory of the Slovak Republic and Czech Republic. It has been shown that distilled water is a good medium for collecting the post-explosion traces of nitro compounds (TNT and DNT) in testing ground. The separation, purification and simultaneous concentrating of aqueous extracts of post-explosion residues was realised by means of SPE (the extraction discs type C18). The procedure is simple and allows obtaining extracts for organic and inorganic analyses in a single operation, at a considerably preconcentration the traces of organic explosives. The ECD detector was shown to be highly selective and its combination with GC makes it an efficient tool in trace analysis of organic components of post-explosion residues. Similarly, also ion chromatography (IC), thanks to its sufficient sensitivity, is well applicable to trace analysis of inorganic components in these residues.

Introduction

The aim of analysis of post-explosion products is to determine the type of explosive material that exploded on the scene of the crime (bomb attack, non-professional production etc.). This task is considerably complicated by the number of unfavorable factors: trace quanta of non-reacted explosives are analyzed which are dispersed to a radius of several meters and are accompanied by interfering impurities in amounts that are orders of magnitude higher. High demands are the collecting of the traces, their extraction and purification of the extracts as well as the highly sensitive and relevant analytical evidence.1 One of the best media used for collecting of the traces is preferably water,2 which in contrast to organic solvents dissolves only a negligible part of the organic component of the explosive depending on its solubility at the given
Another advantage of water as compared with organic solvent is the fact that it is environmentally friendly.\cite{3}

One of the simplest but also most efficient pre-separation techniques, which are used in analyses of aqueous solutions, is solid phase extraction (SPE).\cite{4} It can be used also in the case of analyses of the post-explosion extracts.\cite{5} An experiment of this type was described by Thompson et al.\cite{3} Tontarski et al.\cite{6} found out that organic explosives (RDX, PETN and TNT) are best sorbed on non-polar sorbents such as C\textsubscript{18}.

A procedure of purification of post-explosion products (removing polar impurities that catalyse thermal decomposition of PETN) for gas chromatography was described by Kolla.\cite{7} The above-mentioned complexity of analysis of post-explosion products necessitates application of at least two independent analytical methods in order to get relevant results. These methods-used for organic component-involve, e.g., HPLC/TEA, LC/MS, GC/ECD, GC/TEA, GC/MS. For the inorganic components it is possible to use colour reactions or ion chromatography.\cite{1}

Gas chromatography (GC) is frequently applied to investigation of traces of explosives in the post-explosion extracts. Improvements of capillary columns have brought an increase in applicability of GC to analyses of thermally instable explosives (liquid nitrate esters and nitramines).\cite{8}

The majority of industrial explosives (e.g. dynamites) contain liquid nitrate esters belonging among thermally instable secondary explosives.\cite{9} Besides the active basis of dynamites (which is blasting gelatine: 8% NC and 92% NG), they contain oxidizing agents (e.g. ammonium nitrate) nitroaromatic compounds (DNT and TNT), wood flour, nitrocellulose, dyestuff, trace additives that are specific for individual manufacturers. Determination of wood flour and dyestuff by gas chromatography is out of question because their transformation into vapors is impossible.\cite{10}

For dynamites it is characteristic the presence of NG and EGDN; however, these components are hardly to analyzed because they are evaporated due to the high explosion heat. The components having higher vapor pressures partially disappear when the post-explosion gases are condensed on cold surfaces: therefore, it is better to compare only compounds with similar vapor pressures.\cite{10}

At present, dynamite explosives can be detected very selectively thanks to the nitroaromatic compounds (most frequently DNT and TNT) presented in slight amounts within the complex composition of extracts of post blast residues. The carcinogenicity of nitroaromatics (e.g. 2,6-dinitrotoluene) has led to their gradual replacement by less toxic but harder analysed explosives.\cite{10}

The applicability of GC-ECD to analyses of post-explosion products is reported by McCord & Bender.\cite{11} Practical applications in this area are described by Kaplan &
ZITRIN.\textsuperscript{12} As the ECD detector becomes easily contaminated, the GC-ECD method can only be used for analyses of relatively pure extracts. Its selectivity makes it possible to analyse organic secondary explosives containing nitro groups. The application of ECD is also very important in the identification of secondary explosives presented in matrices contaminated with hydrocarbons.\textsuperscript{13}

Ion chromatography (IC) possesses sufficient sensitivity to be favourably applicable to trace analysis of post-explosion product of industrial secondary explosives existing in ionic forms.\textsuperscript{1} Its complex utilisation in analyses of cations and anions in samples of industrial secondary explosives as well as their post-explosion residues was reported by REUTTER et al.\textsuperscript{14}

The verification of GC-ECD and IC in the applications for specification of post-explosion residues of secondary explosives most frequently abused on the territory of the Slovak Republic and Czech Republic (TNT and Danubit 2) is dealt with in M.Sc. Thesis.\textsuperscript{15} This present paper reports the most important findings of the said Thesis.

**Experimental**

The most frequently misused secondary explosives on the territory of CZ and SR involve TNT and the dynamite explosive Danubit 2. These two products were used in Thesis\textsuperscript{15} for verification of analytical techniques of the post-explosion residues. The secondary explosive Danubit 2 is composed of ethylene glycol dinitrate (EGDN), glycerol trinitrate (NG), cellulose nitrate (NC), dinitrotoluene (DNT), ammonium nitrate (AN), sodium nitrate (SN), wood flour, carboxymethylcellulose, micro-ground limestone and ferric oxide (as a dyestuff).

**Apparatus and instruments**

**Apparatus for SPE**

For the purification and pre-concentrating of post-explosion products were used Empore 3M octadecl C\textsubscript{18} extraction discs, which are suitable for environmental analyses. The SPE disc was attached to the grid of extraction apparatus, and the sample was sieved through the SPE disc, the necessary underpressure was created by a water-jet pump.

**Apparatus for analyses of extracts**

**Gas chromatograph with electron capture detector (GC-ECD)**

The standards of explosives (TNT, DNT, NG a EGDN) and the SPE extracts obtained were analysed on a Hewlett Packard 5890 chromatograph, using a capillary column HP-5 of 5 m length, 0.53 mm inner diameter, and 0.88 \(\mu\)m film thickness.
An electron capture detector (ECD) was used for the detection, and the carrier gas was nitrogen of 20 kPa overpressure. The sample was injected directly (on-column), the injector temperature being 70 °C. The temperature regime was as follows: the column was kept at 70 °C for 1 min, whereupon the temperature was increased at a rate of 15 °C/min to 150 °C, and 5 °C/min to 230 °C, and this final temperature was kept 8 min.

All the GC-ECD chromatograms measured were evaluated by means of CHEMSTATION program.

**Ion chromatograph**

The aqueous extracts filtered through solid phase (SPE) were submitted to analysis by means of an ion chromatograph Dionex DX-20. The analysis of cations took place in a system consisting of a pre-column CG 12A (4×50 mm), a column Ionpac CS 12A (4×250 mm), and a suppressor CSRS-Ultra 4 mm. The system for analysis of anions consisted of a pre-column AG 9-HC 4 mm, a column Ionpac AS 9-HC 4 mm, and a suppressor ASRS-Ultra 4 mm. The elution solvents for cations and anions were 2.2 mM H$_2$SO$_4$ (1 g H$_2$SO$_4$ per 1 l H$_2$O) and 9.0 mM Na$_2$CO$_3$ (18 ml Na$_2$CO$_3$ per 1 l H$_2$O) with flow-rates of 1.00 and 1.07 ml/min at the overpressure of 2300–2400 and 1600 psi (1 psi = 6.89 kPa).

**Field work and sample collecting**

All the experimental explosions were carried out in a stone quarry in Malé Karpaty (Mountains) near Bratislava in Slovakia. The samples used were low-diameter charges of dynamite (Danubit 2) of total masses 200, 400, and 600 g, and tritol charges of total masses 200 and 400 g. The initiation was accomplished by means of Initiator No. 8. The individual charges were placed on the terrain in such a way as to facilitate collecting the post-explosion samples. Also the particular spots of explosions were chosen carefully so as to avoid any contamination from previous explosions. After the detonation, samples were taken from the explosion epicentre and surrounding surfaces of stones near the place of explosion (roughly one and two meters from the explosion epicentre). The traces of post-explosion products were collected by wiping off c. 1 m$^2$ stone surface area with a wet cloth. The extraction was carried out using 700 ml distilled water. The wet cloth with collected traces of explosives was wrung out into a clean bottle. The aqueous extracts of post-explosion products obtained in this way had the volume of c. 200 ml each. The same procedure was used for all the samples.
Preparation of samples for solid phase extraction (SPE)

At first, all the investigated aqueous samples of post-explosion residues from dynamites and TNT were left to sediment. Before proper concentrating the analyte and its simultaneous purification by the solid phase extraction (SPE), the extracts were filtered through a paper filter (blue ribbon) in order to remove mechanical impurities.

Solid phase extraction (SPE)

After preliminary purification of the samples by filtration they were subjected to extraction with solid sorbent C₁₈: 50 ml aqueous extract was left to flow through the extraction disc. Water soluble components of the explosive flowed through the disc while the organic ones remained trapped on it and were subsequently washed out with a small amount of organic solvent (10 ml n-hexane). Details of this separation technique are given elsewhere. The extractions provided organic and aqueous extracts: the former (containing n-hexane) were used for analysis of explosives of organic origin (TNT, DNT, EGDN and NG), while the latter were used for analysis of inorganic water-soluble components of explosives (Na⁺, NH₄⁺, and NO₃⁻ ions).

Results and discussion

Solid phase extraction

Considerable attention was paid to the choice of purification procedure since the electron capture detector (ECD) used is easily contaminated.

As already stated, the separation, purification and pre-concentrating of the analytes from the post-explosion extracts were realised with the help of extraction discs C₁₈. This purification technique was time consuming, and moreover, it was impossible to filtrate equal volumes through the discs. Fine colloid particles collected together with the post-explosion residues during sample collecting could not be perfectly removed by the paper filter. Therefore, the pores of extraction disc get blocked after some time, which also affects the flow rate of n-hexane during washing out the organic extracts from the extraction disc. At a slow flow rate and large underpressure, the n-hexane tends to evaporate rapidly, and the purified organic extracts obtained have then various volumes.

Hence, the drawback of SPE is low rate of processing of analyte solutions if the samples are considerably contaminated. Purification of 50 ml aqueous solution of post-explosion products can take as long as 30–60 min. Advantages of this procedure are its simplicity and the possibility to obtain the extracts for both organic and inorganic analyses simultaneously, the traces of organic explosives being considerably pre-concentrated.
GC-ECD analysis of TNT and Danubit 2 in organic extracts of post-explosion residues

On the basis of literature data and structure of the substances determined, gas chromatography with ECD was chosen for the given analysis. The reason of this choice lies in the high sensitivity of the electron capture detector (ECD) to electronegative atoms (nitrogen compounds) and its low sensitivity to contaminants. The said detector was calibrated by means of standard solutions (2,4,6-TNT, technical DNT, EGDN and NG) with subsequent specification of detection limit for selected types of explosives (see Table 1). The amount of sample injected was low (1 µl). Thermally unstable explosives (EGDN, NG) were analysed with the application of direct injection (on-column) and a short capillary column (5 m).

Table 1. The detection limits for the chosen types of explosives

<table>
<thead>
<tr>
<th>Standard of explosives</th>
<th>Limits of detection of ECD</th>
</tr>
</thead>
<tbody>
<tr>
<td>TNT</td>
<td>0.05 mg/l</td>
</tr>
<tr>
<td>DNT</td>
<td>0.1 mg/l</td>
</tr>
<tr>
<td>EGDN</td>
<td>2.5 µg/l</td>
</tr>
<tr>
<td>NG</td>
<td>5 µg/l</td>
</tr>
</tbody>
</table>

At the above-given conditions of GC-ECD analysis, the analysis time for the investigated group of compounds was up to 6 min. All the purified and concentrated extracts of post-explosion residues from TNT and Danubite 2 were measured at identical conditions of the analysis. The qualitative analysis was based on comparison of retention times of the analytes and standards. The quantitative analysis (concentration) of the individual components in the post-explosion extracts was based on the calibration of detector (the surface areas of peaks). The results of measurements are summarised in Tables 2 and 3.

Table 2. Concentration of TNT in different distances

<table>
<thead>
<tr>
<th>Type of charge</th>
<th>Amount of charge (g)</th>
<th>Place of sampling (m)</th>
<th>Peak Area of TNT</th>
<th>Concentration of TNT in post blast residues (mg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>TNT</td>
<td>200</td>
<td>0</td>
<td>35053620</td>
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<td>1</td>
<td>40562530</td>
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<td></td>
<td>400</td>
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<td>28728040</td>
<td>23.96</td>
</tr>
</tbody>
</table>
Table 3. Concentrations of DNT and EGDN in different distances from explosion of Danubit 2

<table>
<thead>
<tr>
<th>Type of charge</th>
<th>Amount of charge (g)</th>
<th>Place of sampling (m)</th>
<th>Peak Area of DNT</th>
<th>Peak Area of EGDN</th>
<th>Concentration of DNT in post blast residues (mg/l)</th>
<th>Concentration of EGDN in post blast residues (mg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Danubit 2</td>
<td>200</td>
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<td>8464321</td>
<td>457185</td>
<td>14.55</td>
<td>2.69</td>
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<tr>
<td></td>
<td>200</td>
<td>1</td>
<td>51186175</td>
<td>73960</td>
<td>79.83</td>
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<tr>
<td></td>
<td>200</td>
<td>2</td>
<td>31112250</td>
<td>47453600</td>
<td>49.16</td>
<td>35.29</td>
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<td>0</td>
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<td>1369140</td>
<td>105.45</td>
<td>3.33</td>
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<td>1</td>
<td>42370730</td>
<td>1708240</td>
<td>66.36</td>
<td>3.56</td>
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<td></td>
<td>400</td>
<td>2</td>
<td>43039080</td>
<td>250146</td>
<td>67.38</td>
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<td>600</td>
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<td>4198780</td>
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<td>5.67</td>
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</tr>
<tr>
<td></td>
<td>600</td>
<td>2</td>
<td>5401747</td>
<td></td>
<td>9.87</td>
<td>X</td>
</tr>
</tbody>
</table>

The GC-ECD chromatograms of post-explosion products from TNT (Figure 1) showed three peaks identifiable by comparison with the retention times of standards. In the case of TNT charge, the organic extracts always contained TNT in each sample irrespective of the charge mass and the distance of sample collecting from the explosion epicentre (Table 2). The TNT concentration in the post-explosion extracts varied from 6 to 50 mg/l. Besides TNT, also the presence of DNT could be detected, which is typical of technical tritol (DNTs are intermediates in production of TNT). It must be pointed out that TNT for civilian applications usually is of lower purity than military TNT.

The GC-ECD chromatograms of post-explosion products from Danubit 2 exhibited four dominant peaks (Figure 2). The first was identified as the solvent (n-hexane), and the other three were EGDN, 2,4- and 2,6-DNT. In each sample of the organic extracts both the isomers of dinitrotoluene were present. In the case of the 600 g charge of Danubit 2, the result was four times negative for EGDN, see in the samples taken 1 and 2 m from the explosion epicentre (Table 3). The concentrations were in the range from 2 to 100 mg/l for the dinitrotoluenes, and from 2 to 35 mg/l for EGDN. Using its characteristic retention time, NG was not detected at all although it forms-along with EGDN-a component of industrial secondary explosives of dynamite type produced on the territories of CZ and SR. The analysis was not designed to identify other components of Danubit 2 (NC, wood flour, carboxymethylcellulose, dyestuff). Partial failure of identification of EGDN and NG was probably due to their thermal decomposition during explosion or to hydrolysis during sample collecting.
Splitting of organic explosives can proceed in the shock wave or at the contact with surrounding environment (rock) and air moisture. The samples of aqueous extracts of post-explosion residues before SPE were kept in refrigerator at lowered temperature in order to minimise the losses due to hydrolysis. Since the extraction of traces of explosives was performed with water, the results of analysis of the components followed could be partially affected by hydrolysis during the period of time between sample collecting and subsequent sample purification by SPE.

![GC-ECD chromatogram of post-explosion analysis of TNT](image)

Figure 1. GC-ECD chromatogram of post-explosion analysis of TNT
R. VARGA, U. PAVOL: Post-explosion residues

Figure 2. GC-ECD chromatogram of post-explosion analysis of Danubit 2

Analysis of cations and anions in aqueous extracts of post-explosion residues from Danubit 2

The portion of aqueous extracts from Danubit 2 obtained from SPE was analysed for the presence of inorganic components by means of IC. The results are summarised in Table 4.

Table 4. Concentrations of cations (Na\(^{+}\) and NH\(_4\)^{+}\) and anion NO\(_3\)^{−}\) in different distances from Danubit 2

<table>
<thead>
<tr>
<th>Type of charge</th>
<th>Amount of charge (g)</th>
<th>Place of sampling (m)</th>
<th>Concentration of Na(^{+}) (mg/l)</th>
<th>Concentration of NH(_4)^{+} (mg/l)</th>
<th>Concentration of NO(_3)^{−} (mg/l)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Danubit 2</td>
<td>200</td>
<td>0</td>
<td>1.94</td>
<td>3.59</td>
<td>194.48</td>
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<tr>
<td></td>
<td>200</td>
<td>1</td>
<td>7.06</td>
<td>5.14</td>
<td>35.91</td>
</tr>
<tr>
<td></td>
<td>200</td>
<td>2</td>
<td>7.55</td>
<td>2.12</td>
<td>15.38</td>
</tr>
<tr>
<td></td>
<td>400</td>
<td>0</td>
<td>4.10</td>
<td>8.49</td>
<td>439.93</td>
</tr>
<tr>
<td></td>
<td>400</td>
<td>1</td>
<td>21.39</td>
<td>14.33</td>
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<td>9.62</td>
<td>10.63</td>
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<tr>
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<td>600</td>
<td>0</td>
<td>2.46</td>
<td>6.58</td>
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<tr>
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<td>600</td>
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<td>8.00</td>
<td>8.30</td>
<td>76.25</td>
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<td></td>
<td>600</td>
<td>2</td>
<td>12.93</td>
<td>20.17</td>
<td>201.71</td>
</tr>
</tbody>
</table>
Since ammonium nitrate forms a basic component of Danubit 2, predominant attention was paid to the NO$_3^-$ anion. Besides Na$^+$, NH$_4^+$, Ca$^{2+}$ and NO$_3^-$ ions (see Figures 3 and 4), which are components of this dynamite, also other ions were found in the samples. Among the most significant ones there were K$^+$, Cl$^-$, PO$_4^{3-}$ and SO$_4^{2-}$ (see Figures 3 and 4). In the cases of potassium, chloride, phosphate and sulphate, the presence is due to contamination of real samples by ions from environment. The presence of nitrites can be interpreted by reduction of the oxidising agents (NH$_4$NO$_3$ and NaNO$_3$) during the explosion. The ions detected in the post-explosion extracts were Na$^+$, NH$_4^+$ and NO$_3^-$. The concentrations varied from 2 to 20 mg/l for Na$^+$ and NH$_4^+$ cations and from 15 to 490 mg/l for the NO$_3^-$ anion.

Figure 3. IC chromatogram of cations at real samples of post blast residues of Danubit 2. Peaks: (1) Na$^+$, (2) NH$_4^+$, (3) K$^+$, (4) Mg$^{2+}$ and (5) Ca$^{2+}$

Figure 4. IC chromatogram of anions at real samples of post blast residues of Danubit 2. Peaks: (1) F$^-$, (2) Cl$^-$, (3) NO$_2^-$, (4) NO$_3^-$, (5) PO$_4^{3-}$ and (6) SO$_4^{2-}$
Distribution of post-explosion residues

A very important factor is correct determination of the place with the most probable occurrence of traces of explosives. The knowledge of distribution of post-explosion residues forms a basis of criminological expertise of attacks with explosives. Therefore, samples were taken at various distances from the epicentre of explosion, the trapping surfaces being located perpendicularly to the direction of expansion of gases. In order to map the distribution of particles, suitable experimental arrangement was adopted with metal plates of 1 m² surface area located at various distances from explosion epicentre, allowing determination of the amount of explosive per 1 m² at a given distance. This experiment was designed to find approximate distribution of non-reacted particles in the determination of sensitivity of GC-ECD and IC for trace analysis of post-explosion products.

The values measured (see Tables 2, 3 and 4) were plotted in diagrams showing the dependence of amount of post-explosion residues upon the distance from the explosion epicentre (see Figure 5a – DNT, Figure 5b – TNT, Figure 6a – NH₄⁺, Figure 6b – NO₃⁻). All the diagrams show the distribution of non-reacted particles of original explosives (TNT and Danubit 2) at various distances.

The dispersion of post-explosion residues is a very complicated problem, because the distribution of these particles during explosion is affected by a number of unfavourable factors (first of all mass, type of the explosive used, and direction of initiation). The probability of occurrence of non-reacted particles increases with decreasing efficiency of the secondary explosive, with its decreasing mass, and with decreasing diameter of its charge.

In the case of detonating secondary explosives, the non-reacted particles are accelerated by the expansion of the gases formed, in the final phase of the flight they could to be affected by wind. The particles are heated by the shock wave, and in some cases they can have the form of droplets of melt, which subsequently solidify on cold surfaces of the surrounding environment. The non-reacted particles of explosives are present in very slight quanta in the vicinity of the explosion, being irregularly dispersed on a large surface from the explosion epicentre.
Figure 5. Amounts of DNT (Fig 5a) and TNT (Fig 5b) in different distances

Figure 6. Amounts of cations NH₄⁺ (Fig 6a) and anions NO₃⁻ (Fig 6b) in post-explosion extracts of Danubit 2

**Conclusion**

The study results have shown that distilled water is a good medium for collecting traces of nitro compounds (TNT and DNT) because it does not extract any large amounts of undesirable impurities (it provides the purest extracts). Another advantage of water is the possibility to avoid organic solvents with concomitant posing a lower ecological load on the environment. However, the application of water can also bring considerable complications in analyses of nitrate esters (NG, EGDN) due to their hydrolysis, which can be partially eliminated by keeping the extracts at low temperatures or in dry state or-best of all-by quick processing of the extract.
The separation, purification and simultaneous pre-concentrating of the explosives collected in water from the post-explosion extracts were carried out by solid phase extraction (extraction discs of C$_{18}$ type). This procedure is advantageous in being simple and allowing extracts to be obtained for both organic and inorganic analyses at the same time, the traces of organic explosives being considerably pre-concentrated. At present, SPE represents a dynamically developing separation technique and is more and more often adopted in analyses of post-explosion products.

The results of measurements confirmed the high selectivity of ECD detector, its combination with GC being effective in trace analysis of organic components of post-explosion products. The advantages of GC are in the speed of analysis and simplicity of measurements.

The analysis of aqueous extracts was carried out by ion chromatography (IC), which managed to prove the presence of the cations (Na$^+$, NH$_4^+$) and anion (NO$_3^-$) followed. It was confirmed that IC, thanks to its sufficient sensitivity, is well applicable to trace analysis of inorganic components of post-explosion products.

The experiments carried out showed that the tested dynamite (Danubit 2) can be detected in post-explosion products by the presence of DNT (GC-ECD) and Na$^+$, NH$_4^+$ and NO$_3^-$ ions. A more precise identification of this type of dynamite can also make use of the occurrence of EGDN in its post-blast residues. The monitoring of distribution of the non-reacted particles showed that collecting of traces of explosives for reliable detection is sufficient as far as the distance of 2 m from the explosion epicentre.

With regard to the complexity of both the matrix and the analyses it is impossible to develop a simple method for analysis of traces of explosives in post-blast residues.

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References


