



Chemical analysis of post explosion samples obtained as a result of model field experiments



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ABSTRACT

Five different explosives were detonated in a series of field experiments. Each experiment (detonation of the charge of each specific explosive) was repeated three times. The experiments were conducted under controlled conditions, exceeding those of research published so far. Detonated charges were uniform in size and, as far as possible, in shape. The explosives used originated from the same batch. Additionally, the same kind of electric detonators were used. Witness plates (sheets of galvanised steel 100 cm × 90 cm × 0.5 mm) were used to collect post-blast residues in a reproducible way. They were placed relatively close to the charge to minimise the influence of the wind. Samples were collected by systematic swabbing of the surface of the plate by acetone moistened cotton swabs. Samples were packed tight, transferred to the laboratory, and extracted with methanol. Extracts were concentrated by solvent evaporation, cleaned by centrifugation, and analysed using HPLC-DAD. Each extract was analysed three times and the mean value of the amount of the given explosive within the extract was calculated. For each of the explosive materials used the results of the repetition of the experiments proved them to be irreproducible. After each detonation of a specific charge different amounts of given explosives were found in post-blast samples. Also, the intuitively expected relationship between the distance from the charge and amount of post-blast residues were not observed. These results are consistent with previously published results of field experiments. The lack of reproducibility may be explained by differences in efficiency of detonation. The efficiency of a detonation may be influenced even by small differences in the shape of the charge as well as by the position and properties of the detonator. The lack of dependency between the amount of the explosive in the post-blast samples and the distance from the charge may be explained by the fact that during detonation, particles of unreacted explosives are not uniformly dispersed in all directions.

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1. Introduction

The detection of explosive materials at a crime scene enables investigators to distinguish between an explosion caused by an explosive as opposed to, for example, explosions caused by over pressurisation of hydraulic systems or accidental ignition of fuel-air mixtures (flammable gases, vapors of flammable liquids, or dust mixed with air). Information about what kind of explosive was used also helps to establish its possible source and thus to identify the perpetrator. The procedures of sample preparation for different types of materials as well as the parameters of chromatographic analysis are published and available [1]. Nevertheless, there are still many problems to overcome. The most difficult problem concerns representative sampling and the consequent interpretation of analytical results. Detonation is a complex phenomenon that is

likely to be affected by the kind, size, and shape of the main explosive charge, along with the position and orientation of the detonator, and even the weather conditions at the time of detonation. To better understand those phenomena some authors conducted field experiments [2–11]. To collect the post explosive samples in a repetitive way and to eliminate contamination from previous detonations different approaches were utilised: detonation of the charges suspended above a layer of fresh snow or snow covered ice [4–7], using trays placed on the ground or a plastic tarp [5,7,8], or using witness plates placed vertically, and perpendicularly to the direction of the shock wave propagation [2,3,9–11].

Surprisingly, for all of the published research the results of experimental repetitions proved to be unrepeatable as different amounts of explosive residues were detected after each detonation in the same experimental setup [4–8]. The differences in the calculated amounts of given explosives remaining after detonation were very high and ranged over a few orders of magnitude, even if only high-order detonations were taken into account [4–6]. Furthermore an expected correlation between the amount of detected

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explosives and distance from the detonated charge was not confirmed by experimental data [2–5,7].

Another expected correlation between the size of the charge (amount of explosive before detonation) and the amount of explosive within post explosion samples was also not confirmed by the results of field experiments [4,7].

The lack of reproducibility for detonation experiments is most often explained by the fact that the amount of unreacted explosive depends on the efficiency of the detonation. This can be affected even by small and random changes in the properties of an explosive, the shape of the charge, or the properties and position of detonator [2]. The same factor is most probably responsible for the lack of correlation between the size of the charge and the amount of explosive in the residues. Bigger charges can leave less residues compared to smaller ones if the detonation was more efficient.

The lack of correlation between the distance from the charge and the amount of explosive results, most probably, from the problems with representative sampling—particles of unreacted explosive which are dispersed by detonation are not homogenous in size and shape. Some bigger pieces being heavier can travel longer distances and if by chance they are sampled they can have influence on the results [2,4,5,8]. Another important factor which can influence the direction of the expansion of post explosion residues (and thus affect the results) is wind. The influence of wind was clearly confirmed by the results of detonations on snow. The soot darkened area was never circular but always skewed by the wind [4–6].

As it can be seen, there are many possible factors which can influence the amount and distribution of post explosive residues. The results of previously published research indicate that an explosion is a highly unreproducible process both for amount, and distribution of explosives residues. Nevertheless these conclusions cast doubts because they were drawn from limited data. Such limitations include: reliance of data from single experiments, or the experimental setup was not uniform for consequent experiments—charges of different sizes, shape, variations in the position of the detonator or differences in the booster charge utilised. Additionally, only one type of explosive material was used.

The aim of the presented research was to clarify the aforementioned doubts by conducting more extensive field experiments and in a more stringent and controlled way than previously published research. Uniform charges of explosives were detonated three times in the same experimental setup. Witness plates were situated close to the charges to eliminate the influence of wind.

The experiments (three detonations in the same experimental setup) were conducted for five of the most common explosives: TNT, RDX, PETN, dynamite, and Composition B.

2. Materials and methods

2.1. Materials and apparatus

Analysis was conducted using a liquid chromatograph with a diode array detector (LaChrom Elite, VWR-Hitachi, Radnor, Pennsylvania, USA) equipped with a CN column (LiChroCART 250-4; LiChrospher[®] 100 CN 5 μ m, Merck, Darmstadt, Germany). The capacity of the sample loop was 20 μ l. Gradient elution was conducted with a mixture of water, methanol, and acetonitrile (all solvents of gradient grade for liquid chromatography by Merck). The mobile phase was pumped at 1 mL/min. Each run lasted for 30 min. The equilibration time was 5 min. The analytical wavelength used for the calibration and analysis was 205 nm. The analytical signals (y), as peaks areas in arbitrary units, were measured using EZChrom Elite 3.1.7, Scientific Software Inc. The program of elution is

Table 1

The program of gradient elution for HPLC-DAD analysis. The mobile phase was pumped at 1 ml/min. The last two rows (30–35 min) describe the equilibration step.

Time [min]	Methanol [%]	Water [%]	Acetonitrile [%]
0	5	85	10
5	5	75	20
25	65	15	20
26	80	0	20
30	80	0	20
30.1	5	85	10
35	5	85	10

Table 2

Concentration of analytes in standard mixtures which were used for calibration.

Standard number	EGDN [mg/ml]	NG [mg/ml]	RDX [mg/ml]	TNT [mg/ml]	PETN [mg/ml]
1	0.0465	0.0465	0.0461	0.0471	0.0466
2	0.0326	0.0326	0.0323	0.0330	0.0326
3	0.0233	0.0233	0.0231	0.0236	0.0233
4	0.0140	0.0140	0.0138	0.0141	0.0140
5	0.0046	0.0046	0.0046	0.0047	0.0047

presented in Table 1. An exemplary chromatogram obtained for a standard mixture of analysed compounds (standard no. 1, see Table 2) is presented in Fig. 1.

2.2. Calibration, LOD and LOQ

Standard mixtures for the five most common organic explosives (EGDN, NG, PETN, TNT, and RDX) were used for calibration. Solutions of PETN, TNT, and RDX were obtained by dissolving pure substances in methanol (explosives produced by NITROERG, Bierun, Poland). For NG and EGDN, ready standard solutions were utilised: 0.1 mg of NG in ethanol and 0.1 EGDN in acetonitrile (both standards by AccuStandard inc, New Haven, CT, USA). Ready standard mixtures were stored in a fridge, in the temperature 0 °C. The concentration of analytes in standard mixtures are presented in Table 2. Four analyses were conducted for each of the five calibration levels. For each of the analytes the equation of the calibration curve was calculated using linear the regression method. Parameters of the calibration curves described by equation $y=ax+b$ are given in Table 3.

The calculations with application of *t*-Student test were made in the aim to confirm the linearity range, which was determined by minimum and maximum concentrations of the analytes in standard mixtures which were used for calibration. (see Table 2). The following two null hypothesis (H_0) were considered:

- the calculated slope (a) is equal to zero ($H_0: a=0$),
- the calculated intercept (b) is equal to zero ($H_0: b=0$).

In the case of linear dependency $y=ax+b$ the expected result is to reject the null hypothesis for slope and accept the null hypothesis for the intercept.

The limit of detection (LOD) and limit of quantification (LOQ) were also determined for each of organic explosives. Fluctuations of the baselines in the proximity of analytes peaks, caused by unknown co-extracted impurities were measured in the form of peak areas. Ten values of peaks areas were obtained for each of the analytes and they were used to calculate the concentrations, using

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