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A Survey of Background Levels of Explosives and Related Compounds in the Environment*

ABSTRACT: In this paper, swabings from various public areas in 28 different cities throughout the United States were collected. A wide variety of objects were tested, ranging from park benches and telephones to sign posts and mailboxes. Inorganic anions and cations common in low explosives were detected simultaneously using capillary zone electrophoresis. Organic high explosives including nitroaromatics, nitramines, and nitrate esters were detected using gas chromatography with a pulsed-discharge electron capture detector. Confirmation of selected results was performed by ion chromatography and negative ion chemical ionization GC/MS. In general the results provide two major conclusions: (i) with the exception of nitrate, most low explosive oxidizers are rare in the environment; (ii) no organic explosives or significant interferences to these explosives were detected.

KEYWORDS: forensic science, explosives, capillary electrophoresis, gas chromatography, inorganic ions, environmental, electron capture

When performing postblast explosives residue examinations, it is important to ensure that analytical data from swabs taken at the scene of the crime provide unambiguous results. Otherwise, matrix interferences can obscure or obfuscate the presence of residue from the blast. This problem can be addressed by collecting comparison samples near the scene to ascertain the environmental factors that might affect the interpretation of analytical results. For many other situations, there is a need to have general information on background levels of explosives in the environment. This information can be utilized in policy decisions, such as determining alarm levels in airport screening and setting threshold levels for explosive residues on hands and documents. However, only a few papers have been published on background levels of explosives and interfering compounds in the environment (1–4).

The issue of background interferences has become more compelling over the past few years, particularly in the forensic analysis of large and complex crime scenes such as the 1993 World Trade Center (WTC) bombing and the Pan Am crash in Lockerbie, Scotland. For example, the WTC bombing occurred in a garage which contained a complex matrix of background compounds both at the seat of the blast and in its surrounding environment. Broken sewage pipes, concrete dust, and deicing salts were all present. These materials complicated the process of distinguishing the components of postblast residue from the overall sample matrix. Similar situations can occur at other bombing crime scenes.

Data regarding the environmental background levels of constituents found in explosives is needed for the proper analysis and interpretation of evidence. For example, elevated levels of organic

nitrates might be expected in locations such as bathrooms where perfumed cleansers containing these compounds are used while at other locations the levels of such compounds may be much lower. Ions such as nitrate and ammonia can be indicators of specific types of inorganic explosives; however, they are also present in fertilizers. Thus it is necessary to try to establish relative levels of explosives and their interfering compounds in the environment to help determine the significance of the results obtained from postblast samples. Then the examiner can better evaluate data and determine if an elevated level is from a known background source or the product of an ex from crime scene samples, the relevance of explosives residue data in court may prove difficult to ascertain. The goal of this paper is to provide a reference study on background levels of explosives in the environment to assist the forensic community and to assist in the interpretation of crime scene data.

Research into background levels of explosives has been performed previously in the United Kingdom by scientists at Defence Science and Technology Laboratory (DSTL). Data on levels of both organic and inorganic explosives in environmental wipe samples were reported (1–3). The Federal Bureau of Investigation (FBI) also reported preliminary results of an explosive residue field survey in the U.S. (4). These studies were useful in establishing that certain ions are ubiquitous in some areas and can potentially interfere in the interpretation of analytical results. However, the studies in the U.S. were limited in sample diversity and location.

The goal of this work was to provide a wider selection of target areas and locations for the determination of the levels of explosives and related compounds in the continental U.S. and Puerto Rico. The data in this study was acquired with the assistance of the FBI. FBI Evidence Response Teams (ERT) in various field offices across the U.S. collected samples from a variety of objects and locations in 28 different cities. The swabs were then analyzed in our laboratories for the presence of trace levels of inorganic and organic compounds which might interfere in explosives analysis. This paper provides the results of these analyses.

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Materials and Methods

Chemicals

The capillary electrophoresis buffer and standards used in this study were prepared using 18.2 M Ω deionized water. The buffer contained 15 mM α -hydroxyisobutyric acid (HIBA), 6 mM imidazole, and 4 mM 18-crown-6 ether obtained from Acros (Morris Plains, NJ), 3 mM 1,3,6-naphthalenetrisulfonic acid (1,3,6-NTS) (Aldrich, Milwaukee, WI), and 5% (v/v) acetonitrile (Fisher Scientific, Pittsburgh, PA). The pH was adjusted to 6.5 using 0.5 M tetramethylammonium hydroxide (TMAOH) (Sigma, St. Louis, MO). Full details of this procedure have been reported previously (5). HPLC grade acetone (Fisher Scientific) was used for all organic extractions.

Standards

Inorganic standards were initially prepared at a concentration of 1800 $\mu\text{g}/\text{mL}$ and diluted to a working concentration of 10 $\mu\text{g}/\text{mL}$. The standards were prepared from analytical reagent grade potassium salts of chloride (Spectrum, Gardena, CA), nitrite nitrate, perchlorate, chlorate (Aldrich), and thiocyanate and cyanate (Acros); analytical reagent grade sodium salt of sulfate (Aldrich); and analytical reagent grade chloride salts of potassium (Spectrum), ammonium, sodium, calcium and magnesium (Fisher Scientific).

For the organic residue analysis, standard solutions of ethylene glycol dinitrate (EGDN), 4-nitrotoluene (4-NT), nitroglycerine (NG), 2,6-dinitrotoluene (2,6-DNT), 2,4-dinitrotoluene (2,4-DNT), 2,4,6-trinitrotoluene (TNT), pentaerythritol tetranitrate (PETN), cyclotrimethylenetrinitramine (RDX), cyclotetramethylenetrinitramine (HMX), and 2,4,6-trinitrophenylmethylnitramine (Tetryl) were obtained from Cerilliant Corporation (Round Rock, TX). The EGDN standard had a concentration of 100 $\mu\text{g}/\text{mL}$, while all the other standards were at 1000 $\mu\text{g}/\text{mL}$ in acetonitrile. A standard mixture was prepared by mixing the 10 compounds at an equal concentration of 10 $\mu\text{g}/\text{mL}$, except for HMX, which was used at 100 $\mu\text{g}/\text{mL}$. Working standards covering the range of 5–500 ng/mL were obtained by serial dilution of the original mixture in HPLC grade acetone (Fisher Scientific, Fair Lawn, NJ).

Samples and Preparation

Sampling was performed using custom prepared kits. Each kit contained a vial with a blank cotton ball (rinsed with deionized water and organic solvent), a pair of gloves, a template, and a pair of disposable tweezers. The evidence collector donned gloves, swabbed the area within the template, and placed the swab into a vial labeled with the date, location, and type of sample. Sampling was performed at various locations including: automobile seats, airports, hotels, office buildings, and many others. Collection occurred during the months of December through June. Figure 1 shows a map of the locations of each city where samples were taken. Negative controls prepared in the FBI laboratory were also included with each kit. Additionally, a set of positive controls were also prepared.

The completed sample kits were then sent to Ohio University for analysis. Cotton swabs were first cut in half with scissors cleaned with ethanol prior to analysis. One half of the cotton ball was prepared for inorganic analysis by placing it into a 15 mL conical vial and extracting with 1.0 mL of 18.2 M Ω deionized water and centrifuging for 8 min at 1000 rpm. The extract was diluted as necessary for analysis. Most samples were run one time; however, samples from 10 cities were run in triplicate. For these samples



FIG. 1—Map of sampling locations in the continental United States. Pre-rinsed dry cotton swabs were used to collect wipe samples from various locations in each city. A location in Puerto Rico, not shown, was also sampled.

relative standard deviations for the common ions Cl and K were calculated to be 0.1–40% for chloride (median 3%) and 4–60% for potassium (median 18%). Larger RSD values typically resulted from low level samples.

The other half of the cotton swab was placed in a disposable plastic syringe to which 2 mL of HPLC grade acetone was added. The solution was expelled through a 0.45 μm PTFE syringe filter (Fisher Scientific, Pittsburgh, PA) and divided into separate 1.0 mL samples. One of the samples was concentrated to approximately 200 μL with a flow of nitrogen for GC analysis, while the other sample was kept as a duplicate for later analysis.

Capillary Zone Electrophoresis

A Beckman Coulter P/ACE MDQ Capillary Electrophoresis System (Fullerton, CA) equipped with a photodiode array (PDA) detector was used for simultaneous analysis of cations and anions (5). Fused-silica capillaries (Polymicro Technology, Phoenix, AZ) of 40.2 cm \times 50 μm I.D. (effective length) were used. From the cathode, the capillary length to the detector is 30 cm and 10.2 cm from the anode. Samples were introduced into the capillary via hydrodynamic injection with injection on the long end for 7 sec and 5 sec on the short end. Indirect UV absorbance detection was performed with dual wavelength detection. Cations were detected at 208 nm, while anions were detected at 235 nm. Baseline separation of all species was attained using a field strength of 248 V/cm at ambient temperature. Pooled standard deviation values for the method migration time repeatability for the standard ranged from 0.007 to 0.013 min for cations and 0.013 to 0.035 min for anions. Detection limits ranged from 1 to 15 $\mu\text{g}/\text{mL}$ for all ions with the exception of sodium (40 $\mu\text{g}/\text{mL}$) which was higher due to an interfering system peak. For this reason, the percentage of samples positive for sodium is likely higher than that listed, however, this ion is of limited interest in explosives analysis because most oxidizers in explosives are present as the potassium or ammonium salt.

Electron Capture Detection

Organic explosives standards and cotton swab extracts were analyzed using a pulsed-discharge electron capture detector (PDECD) coupled to a 6890 GC (Agilent Technologies, Santa Clara, CA) (6). The detector was a PD-D2 model (VICI, Houston, TX)

controlled by a PD-C controller unit, also from VICI. To produce low energy electrons in the electron capture cell, a 3% mixture of xenon in helium (American Gas Group, Toledo, OH) was used as the dopant gas; it was introduced at a flow rate of 3.8 mL/min into the helium plasma. The plasma was generated by a 30 mL/min flow of ultra-high purity helium (Airgas, Parkersburg, WV) passed through a high voltage pulsed electric discharge. The column was an RTX-5, 15 m, 0.25 mm ID, 0.25 μm film thickness (Restek Corp., Bellefonte, PA). The flow rate through the column was kept constant at 2.0 mL/min with a split ratio of 10:1. The injector was kept at a constant temperature of 200°C and the detector at 250°C. The oven temperature program was initiated at 100°C for 1 min, followed by a ramp at 30°C/min up to 230°C, where it was held for 4.67 min. A 1.0 μL injection volume was used for all analyses with an HP 7683 Series Injector (Agilent Technologies). The gas chromatograph was controlled using Chemstation software from Agilent Technologies and the data acquisition was performed using EZChrom version 6.7 from Scientific Software Inc. (Pleasanton, CA) Detection limits for most explosives were under 5 ng/mL with the exception of NG and HMX which were 25 and 50 ng/mL respectively, Table 2.

Results and Discussion

Sample collection was performed by sending sampling kits to investigators in cities across the U.S. In all, 28 cities were sampled across the continental U.S. and Puerto Rico. The individual samples were collected by swabbing an area equivalent to the supplied template (670 cm^2) with a dry cotton swab. The swabs were then packaged and sent to the laboratory for analysis. The exact locations and objects sampled were at the discretion of the investigators, but general sites such as post offices, automatic teller machines, public buildings, and sports arenas were selected.

In the laboratory, cotton swabs were cut in half with an aqueous or acetone extraction performed on each half swab to detect either inorganic or organic explosives respectively. The instrumentation used for screening, capillary electrophoresis, and gas chromatography are commonly found in laboratories performing explosives residue detection; however, modifications were made to the general techniques in an effort to improve efficiency and throughput. In particular, the capillary electrophoresis system used a dual opposite injection to simultaneously detect anions and cations resulting from inorganic explosives residue (5) and a highly sensitive pulsed-discharge electron capture detector coupled to a gas chromatograph was used to screen for organic explosives (6). Complete details on the operation of these instruments have been published in separate papers (5,6).

A subset of those samples producing positive results by capillary electrophoresis was also analyzed by ion chromatography (7). In addition, 10–12 organic extracts which produced peaks with elution times near those of the explosives were examined by liquid chromatography/mass spectrometry (GC/MS) (8). Another subset of samples was screened using GC/MS with negative ion chemical ionization detection (9).

Inorganic Results

The data from different cities was tabulated, and the samples were then grouped into 16 different categories depending on the type of location. The cations analyzed included ammonium, potassium, sodium, calcium, and magnesium. The anions included chloride, nitrite, nitrate, sulfate, perchlorate, thiocyanate, chlorate, and cyanate. The ions that were common throughout the environment

included potassium, sodium, calcium, chloride, nitrate, and sulfate. Ammonium, nitrite, chlorate, and perchlorate were rarely observed.

For example, ammonium was present in 3% of the samples tested. Only 7 of the 16 general locations contained samples that were positive for ammonium, and measured levels were generally below 30 μg . The highest level of this ion, 210 μg , was found on a mailbox in Springfield, IL. In contrast, potassium was found in 44% of all samples measured and in all location categories. For the positive samples, the median level of potassium was 25 μg and the maximum value was 370 μg found on the same mailbox handle in Springfield, IL. Similarly, sodium and calcium were found at all 16 location categories. A swabbing of the front door handle of a town hall in Memphis, TN had the highest level of sodium at 1050 μg and a sample containing 840 μg of calcium was taken from a mailbox in front of a San Antonio, TX hospital. Other high calcium levels included a handrail sample with 390 μg in front of a hospital entrance in San Antonio, and the sample of a mailbox handle in Springfield with 300 μg .

Chloride was found in all location categories and in 58% of all samples. The maximum value was 1100 μg in a men's bathroom sink in the Amerisuites hotel in San Antonio, TX. Other elevated levels included a 1000 μg sample taken from a mailbox in front of a hospital in San Antonio, and a 1000 μg sample recovered from a parking garage in Boston, MA. In general, levels for chloride were moderate with an overall median value of 3 μg . Only six of 167 positive samples contained chloride levels above 50 μg .

Nitrate was also common, showing up in 17% of the samples. It was present in all categories of samples with a maximum level of 110 μg for a mailbox swab taken in front of a hospital in San Antonio. In contrast, nitrite, an ion often found in postblast residue of black powder, was very rare and was detected in only three samples or about 1% of the swabs. The maximum concentration was a sample with a relatively low value of 3 μg from a handrail in a bus in San Juan, PR. These results suggest that determining nitrite would be useful in supporting the presence of nitrate oxidizers in postblast residue due to its relative rarity in the environment.

Sulfate was found in 31% of the samples and in all 16 location categories with a maximum level of 1900 μg on a mailbox in front of a hospital in San Antonio. In San Antonio, a sample of a post at a Greyhound bus terminal had a maximum value of 370 μg , a sink in the men's restroom of the Amerisuites hotel had 460 μg , a handrail at an emergency room of a hospital had 120 μg , and a parking garage in Boston had 200 μg of sulfate ion. All other samples were below 86 μg .

The chlorine-based oxidizers were found to be quite rare. Perchlorate was found in only 1% of the samples and in only three of the 16 location categories with a maximum level of 15 μg on a handrail swab outside a hospital in San Antonio. Chlorate was also found in only 2% of all samples and in only five of the location categories with a maximum level of 19 μg found on a handrail swab outside the emergency room of a hospital in San Antonio. Six of the seven positive samples of this ion were 3 μg and below.

Two other ions tested were cyanate and thiocyanate. These ions are found in postblast residue due to the presence of potassium nitrate oxidizers and carbon fuels in low explosives, but they were quite rare in the environment. Thiocyanate was found in only 1% of the samples and in only two of the 16 location categories with a maximum value of 14 μg found on the handrail swab outside the emergency room of a hospital in San Antonio. Cyanate, OCN^- , was found in 4% of the samples and seven of the 16 location categories with a maximum of 72 μg from a concrete flower base at a hotel in Newark, NJ. The median value for cyanate was 12 μg .

TABLE 1—Levels of anions and cations found on swab samples*

Ion	% Positive Samples	Median Level (µg)	Maximum Level (µg)
Chloride	58%	3	1100
Nitrite	1%	1	3
Nitrate	17%	3	110
Sulfate	31%	10	1900
Chlorate	2%	2	19
Thiocyanate	1%	2	14
Cyanate	4%	12	72
Perchlorate	1%	1	15
Ammonium	3%	26	210
Sodium	25%	81	1050
Potassium	44%	25	370
Magnesium	6%	16	51
Calcium	41%	36	840

* Column 1 lists the percentage of swabs that were positive for each ion. Column 2 lists the median level of each ion in µg and column 3 lists the maximum level µg of each ion found. Analysis performed by capillary electrophoresis using dual opposite injection. Data on reproducibility of the procedure is published in ref 5.

Not all wipe samples contained each ion, thus the results in Table 1 were reported as the percent of samples containing each ion and the median concentration of each ion in the samples that were positive. Table 2 provides the number of positive results for each ion sorted by the 16 sample categories. The data illustrate the wide variety of locations sampled and demonstrate that the trends discovered in the overall results also show up in individual locations. Absolute concentrations or averages were not reported in this table. Instead just the percentage of positive samples was recorded. This is because the absolute concentrations of the ions can be expected to vary with the way the individual sampled the surface, the method by which the swabs were cut, and the effect of the matrix on the extraction. However, the overall results clearly demonstrate the relative levels of particular ions that may occur in a background matrix at a crime scene.

Organic Results

Following the validation of the pulsed-discharge electron capture detector for the analysis of explosives (6), the study of background levels of high explosives in various environmental samples was performed. The majority of samples were very clean and had no

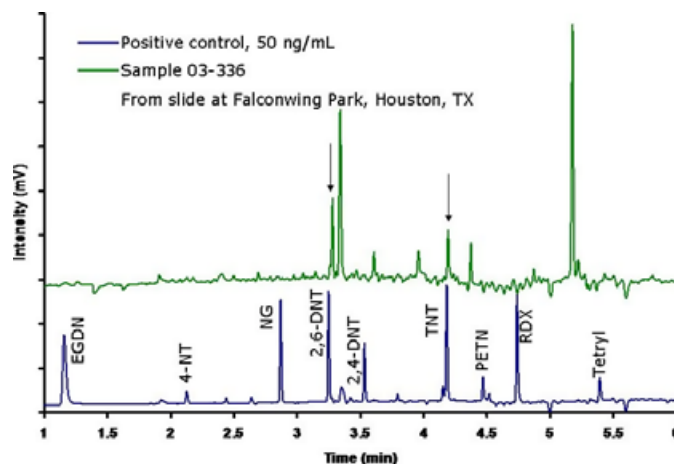


FIG. 2—A comparison of a swab obtained from a children's slide in Falconwing Park in Houston, TX with a 50 ng/mL high explosives standard. The samples were analyzed by capillary GC using pulsed discharge electron capture detection (6). Conditions are listed in the experimental section. Further analysis by LC/MS revealed no explosive materials present in this sample.

relevant peaks distinguishable from the blank controls. Of the total number of samples that were analyzed, only 20 samples showed one or more peaks that were above the background level. None of these peaks had retention times that matched any of the 10 explosive compounds included in this study. By further concentrating the samples, 12 of 333 samples showed indications of explosives; however, at least nine of these results are likely the result of low level interferences present in the solvent. Figure 2 provides an example chromatogram comparing a result obtained from a wipe sample of a children's slide in Falconwing Park in Houston, TX to a 50 ng/mL explosives standard. Further analysis of these samples by LC/MS detected no high explosives.

Conclusions

In this survey, over 300 samples were collected and tested for explosives residue from 28 cities across the U.S. at 16 different location categories. The results yielded base levels of five cations (NH_4^+ , K^+ , Na^+ , Ca^{2+} , and Mg^{2+}) and eight anions (Cl^- , NO_2^- , NO_3^- , SO_4^{2-} , ClO_4^- , SCN^- , ClO_3^- , OCN^-). Nitrate, chloride, and sulfate were detected in a high proportion of samples proving their

TABLE 2—Percentage of positive samples at each location category

Category (# samples)	NH4	K	Na	Ca	Mg	Cl	NO2	NO3	SO4	ClO4	SCN	ClO3	OCN
Auto (9)	0%	44%	44%	56%	11%	78%	11%	33%	33%	0%	0%	0%	22%
Airport (29)	3%	41%	10%	34%	3%	45%	0%	14%	41%	0%	0%	0%	7%
ATM (10)	10%	30%	10%	10%	0%	70%	10%	30%	20%	0%	0%	0%	0%
Government building (28)	0%	61%	29%	50%	0%	36%	4%	4%	14%	0%	0%	11%	0%
Hospital/Hotel (16)	0%	44%	25%	31%	13%	63%	0%	38%	25%	13%	13%	6%	19%
Other indoor location (8)	13%	38%	25%	25%	13%	75%	0%	25%	38%	0%	0%	0%	0%
Mailbox (11)	18%	55%	27%	64%	18%	64%	0%	27%	55%	0%	0%	9%	0%
Money location (6)	17%	67%	33%	67%	0%	50%	0%	17%	17%	0%	0%	0%	17%
Outdoor location (28)	0%	50%	29%	50%	7%	64%	0%	21%	36%	0%	0%	0%	4%
Parking meters (4)	0%	50%	25%	75%	0%	50%	0%	75%	75%	0%	0%	0%	0%
Public building (48)	4%	33%	21%	42%	0%	58%	0%	2%	23%	0%	0%	2%	4%
Post office (13)	0%	62%	54%	62%	23%	77%	0%	8%	38%	0%	0%	0%	0%
Pay phone (14)	0%	36%	21%	43%	7%	71%	0%	21%	36%	0%	0%	0%	0%
Restaurant (16)	6%	50%	19%	38%	6%	63%	0%	6%	31%	6%	0%	0%	6%
Sports arena (23)	0%	48%	35%	35%	4%	43%	0%	22%	26%	4%	0%	0%	0%
Transportation center (23)	0%	26%	17%	17%	9%	70%	0%	26%	43%	0%	0%	4%	0%

ubiquitous nature. Sodium, potassium, calcium, and magnesium were shown to be common ions as well. Other important ions such as nitrite, ammonium, perchlorate and chlorate, cyanate and thiocyanate were shown to be rare.

For organic explosives, the negative results obtained in this study are consistent with data previously published by the Forensic Explosives Laboratory in the United Kingdom (1,3) in 1996 and 2004 using TEA. Both of these studies utilized similar standards and protocols as those reported here. In the U.K. studies more than 670 samples from public areas were analyzed and only eight positive results were obtained, showing trace amounts of RDX (5), NG (2), and 2,4-DNT (1). Overall our results tell the same story; the chances of finding high explosive residues in a public area are minimal.

In general, this study provides investigators with an overview of the background levels of ions in public areas used as markers to detect explosives. The results illustrate the significance of detecting ammonium, nitrite, perchlorate, thiocyanate, chlorate, and cyanate ions on swabs from most crime scenes. These may be considered important markers for the presence of inorganic explosive residue due to their low levels in the environment. More common ions such as nitrate can still be significant but must be interpreted more cautiously. In addition, our results also show that high explosives are extremely rare in most public access areas.

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