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EXPLOSIVES DECONTAMINATION OF
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EXPLOSIVES DECONTAMINATION OF LABORATORY WORKBENCH SURFACES

By

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ABSTRACT

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Instrumental sensitivity mandate that precautions be taken to ensure integrity of explosives evidence after submission to the laboratory for examination. The risk of invalidating potentially compelling explosives evidence by neglecting the possibility of contamination is a concern of the forensic science community, and attention to cross-contamination of cases is of the utmost importance. The laboratory bench decontamination project examines the effectiveness of 409® spray cleaner as a simple decontamination procedure for removal of explosives residues on two types of common laboratory bench surfaces. Bench swab samples were analyzed via gas chromatography / thermal energy analysis (GC/TEA) for both qualitative and quantitative analyses. Samples 1 microliter in size were used in the analysis. Acetone served as a blank and an acetone solution containing 700ng/mL FNT, 750ng/mL NG, TNT, PETN, and RDX, and 75ng/mL EGDN was used as a standard. Results indicate that the use of the spray cleaner alone is not sufficient in the decontamination of benches, and should be coupled with a solvent wipe-down to provide an adequate level of decontamination.

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TABLE OF CONTENTS

LIST OF TABLES

INTRODUCTION

In order for the criminal justice system to function as it is designed to do so, every aspect of the process must be performed as thoroughly and accurately as possible. Ensuring quality in the analysis of forensic evidence is no exception, and an area of serious concern for forensic scientists is in evidence cross-contamination between case materials. The risk of invalidating potentially compelling trace evidence by neglecting the possibility of contamination is a concern of evidence analysts and criminal justice personnel alike.

In light of the recent negative publicity gained by various forensic laboratory agencies, attention to contamination issues is of the utmost importance. However, a literature review indicates that there has not been a considerable amount of written work concerning contamination in forensic work, and very little discussion addressing contamination concerning explosive materials.

Moore, Jackson and Firth[1] carried out a controlled study concerning contamination in a trace laboratory by examining the movement of several types of fibers. While transfer of some types of fibers from one area of a laboratory to another area within the same room was recorded, they concluded that the quality assurances procedures practiced by the laboratory provided a sufficient safety margin to preclude any threat of habitual contamination issues.

Kennedy and Stevens[2] have addressed the issue of contamination as it pertains to a medical context. The use of tracer materials and fluorescent dyes included in simulated patient blood samples revealed widespread contamination throughout the laboratory and

failure of basic hygiene practices. An issue such as this is especially alarming when samples containing the hepatitis B virus or HIV are handled. The study concluded that the use of tracers in a clinical setting is helpful when assessing the quality of hygiene practices and in determining patterns of contamination.

Cook[3] addresses the issue of contamination in a forensic setting from a variety of evidential sources such as fibers, paint, glass, firearms, body fluids in dried stains, including a brief reference to explosive residues. A list of precautions includes cleaning of workbenches, using disposable paper on work surfaces, changing laboratory coats or attire between examinations, and physical separation of space of examinations.

One reference was found that addresses the issue of contamination as it pertains to a trace explosive laboratory. Todd[4] conducts forensic investigations involving the criminal misuse of explosives on the UK Mainland at the Forensic Explosives Laboratory based at Fort Halstead in England. His paper addresses some issues of cross-contamination that have arisen in trials. In particular, contamination does occasionally occur in the trace examinations area of the laboratory; however, the explosives found were of a level less than 10ng, a background level below which no casework results were regarded as significant. In this laboratory, contamination prevention includes dedicating a suite of rooms in which only trace explosives contamination is carried out. Samples come in contact with only disposable surfaces (gloves, disposable glassware and plastic tools), all bench surfaces are first cleaned, then covered with a protective paper barrier before examinations, and monitor samples are analyzed from the laboratory benches, floors and walls on a weekly basis.

While all of these papers and studies address the issue of contamination in a forensic setting of one type or another, none present a set of guidelines that have been tested in order to prevent or control contamination of a workspace in an explosives laboratory setting. This research project will look specifically at the areas of explosives evidence and the potential for and preventative measures against cross-contamination in the form of effective decontamination procedures to be followed between case examinations.

The purpose of the decontamination project is twofold. First is to investigate the ability of a workbench surface to retain high explosives residues. Explosives residues are commonly encountered in a laboratory setting when a solvent extract of a piece of explosives evidence is prepared, when an explosives standard solution is prepared for use in analysis comparisons, or when explosives evidence arrives at the laboratory in the form of a solution. Both a black epoxy resin surface and a white Formica surface were analyzed. The second purpose of the project is to investigate the effectiveness of an ordinary, household spray cleaner (409®) to eliminate traces of explosives residues from the workbench surfaces.

The project was conducted at the Bureau of Alcohol, Tobacco and Firearms (ATF) Forensic Science Laboratory in Rockville, Maryland outside of Washington, DC through an internship opportunity in conjunction with the ATF and Michigan State University. The internship and project were necessary for the completion of the Master of Science in Criminal Justice with Specialization in Forensic Science degree and was carried out over the months of May, June, July and August of 1999. The project was organized into several steps. The first step consisted of a background check of the

fourteen workbenches used in the examination of explosives evidence in the explosives section of the ATF laboratory. The second step involved creating calibration solutions and a calibration program to use in the instrumental analysis of data from the project. The third and fourth steps of the project concerned the deposition and recovery of explosives residue samples on the two types of workbench surfaces in question. The final step addressed the instrumental analysis of the samples and the interpretation of the results. The methods used in the above steps will be explained in detail in the following section.

METHODS

A background check of the workbenches used in the explosives section of the ATF laboratory was performed before the actual decontamination project was initiated. The background check, although not crucial to the project itself, gave an indication as to whether or not low-level contamination of the workbenches was a concern for the laboratory. The background check was performed by swabbing each of the fourteen benches in question with first a dry, sterile cotton gauze pad, followed by a swabbing of a different area of the bench with a sterile cotton gauze pad wetted with 1mL of acetone. The area swabbed on each bench was approximately four inches square. The bench surfaces swabbed were a combination of the two surfaces previously mentioned. The swabs (28 total) were placed in sterile vials containing 3mL of an internal standard acetone solution. The internal standard, also referred to as ISTD, was FNT (fluoronitrotoluene) at a concentration of 700ng/mL. The FNT in acetone internal standard solution was used as an extraction solution throughout the project to monitor the

sensitivity of the instrumental analysis to ensure the instrument was working properly. FNT, a compound not explosive in nature, was chosen as the internal standard since it is easily detected by the instrument used in the analysis, and will not interfere with the explosives' signals. Instrumental analysis was performed with a gas chromatograph / thermal energy analyzer (GC/TEA).

Combining a TEA detector with a gas chromatograph is a useful and popular method of instrumental analysis for nitrogen-containing compounds. As effluent from the chromatograph enters a pyrolyzer, NO_2 is released from organic nitrosyl compounds and converted into NO by a catalytic surface. Pyrolysis products and solvent vapors are removed by a cold trap, and the remaining NO gas reacts with ozone in a reaction chamber. A characteristic infrared chemiluminescent reaction occurs, and its intensity is monitored by an infrared-sensitive photomultiplier tube. The GC/TEA method of analysis for explosive compounds is both sensitive at the picogram (10^{-12}) level and highly selective. The use of a TEA detector coupled with a gas chromatograph in the analysis of explosive compounds has been documented in many instances as being a selective and efficient method. Fine, Yu and Goff[6] found that the TEA analyzer interfaced to a GC was a useful tool for the analysis of explosive residues in a wide variety of forensic as well as environmental applications. The authors found the method of analysis to be simple, rapid, needing little sample preparation or clean-up due to its selectivity, and capable of detecting explosives at low picogram levels from cotton swabs and "real world" samples of post-explosion residues. Douse[6] and LaFleur and Morriseau[7] had similar findings to Fine, et al. using a TEA detector coupled with a gas chromatograph and a high-performance liquid chromatograph, respectively. Hiley[8] states

that GC/TEA analysis of explosives is the principal analytical tool used at the Defence Research Agency, UK.

After the background swabs had been in the extraction vials for four hours, the FNT in acetone solution was extracted from the vials, filtered using a Gelman mini-filter attached to a disposable sterile syringe, and placed in a small, sterile sample vial. The desired extraction time of four hours was determined by creating test samples and leaving them in vials for two, four, and six hours. The two-hour time length did not allow enough of the swabbed materials to be extracted into the solvent, and the six-hour time period gave the same results as the four-hour period. Therefore, the four-hour period was chosen for efficiency. One microliter of each sample was then analyzed via GC/TEA and the results were processed using ChromQuest® software. The FNT in acetone solution was used as a blank throughout the project and was run between each sample and standard. A standard explosives solution was used as the standard throughout the project as well. The standard solution consisted of 750ng/mL of the explosives NG (nitroglycerine), TNT (2,4,6-trinitrotoluene), PETN (pentaerythritol tetranitrate), and RDX (cyclotetramethylene trinitramine), 750ng/mL of the ISTD (internal standard) solution, FNT in acetone, and 75ng/mL of the explosive EGDN (ethylene glycol dinitrate). Parameters for the instrumental analysis are as follows:

GC: 5890 Hewlett Packard Gas Chromatograph
Column: Supelco SPB-5 (5% diphenyl, 95% dimethylsiloxane)
Length: 15 m
ID: 0.32 mm
Film thickness: 0.25 um
Guard column: 36 inches Supelco SPB-5
Temperature program: Initial temperature 35°C
Ramp 5°C/minute to 200°C
Column head pressure: 10psi, Helium

TEA: Thermo Electron Corporation, TEA Model 510 Nitrogen Analyzer
Oxygen Flow to Ozonator: 20cc/min
Pyrolyzer Temperature: GC = 800°C
GC Interface Temperature: 175°C

Upon completion of the background laboratory workbench check, a series of calibration solutions were made and a calibration program was created to aid in the analysis of the project data. The calibration solution series consisted of four solutions containing the explosives and ISTD mentioned above (NG, TNT, PETN, TNT, RDX, FTN and EGDN) with the explosives and ISTD having a concentration of 200ng/mL, 300ng/mL, 500ng/mL and 750ng/mL. The exception was in the case of the explosive EGDN whose concentration was 20ng/mL, 30ng/mL, 50ng/mL and 75ng/mL in each of the four solutions, respectively. The four calibration solutions were each analyzed five times using GC/TEA. These concentrations represent a broad scale of the types of solutions that could be encountered in an analytical laboratory setting. The concentration of the calibration solutions were also chosen based upon the initial concentration of the standards supplied by the company from which they were purchased. Throughout the project, the explosive EGDN had a concentration one factor lower than that of the other explosives. This, too, was due to the initial concentration of the supplied standard.

Deposition and recovery of the deposited materials were performed on a white, Formica bench surface and a black epoxy resin surface. These two bench surfaces are representative of the types of surfaces found throughout the explosives laboratory. The two particular benches used in the deposition step of the project were chosen arbitrarily. Before deposition, the test surfaces were thoroughly cleaned with soap and water, followed by a cleaning with 409®. The deposition on the benches was performed by first creating test tiles by taping off 20 squares approximately four inches square on each bench (40 tiles total). The area of four inches square was decided upon by the area of the bench available to dedicate to the project, and by the number of trials that were deemed necessary to give the desired amount of data with which to work. To purposely contaminate the surfaces, 1mL of an explosives solution containing 1000ng/mL of NG, TNT, PETN, and RDX, and 100ng/mL of EGDN was applied to each tile. This concentration is on the high end of the scale of solution concentrations that would be encountered in the laboratory. It was determined that the deposition solution should be made more concentrated than what would be typically encountered in the lab to truly determine if the resulting decontamination measures are adequate for the spills or contamination situations that are commonly encountered.

The explosives from ten tiles from each surface (20 tiles from the total 40) were then recovered immediately upon drying of the deposited sample. The recovery was performed in the same manner as the background swabbing of the laboratory benches in that a sterile, cotton gauze pad was wetted with 1mL acetone and each tile was swabbed. The swab was then placed in a sample vial containing 3mL of the ISTD solution, FNT in

acetone. After four hours, the solvent was extracted, filtered, and placed in a new sample vial. One microliter of the extract was then used in instrumental analysis.

The remaining ten tiles from each surface were allowed to dry as well, and were then cleaned with 409®. After the application of 409®, the final ten tiles from each surface were then swabbed as the initial tiles were, and extracted in a similar manner. The forty samples were then analyzed via GC/TEA under the parameters described earlier.

RESULTS

Of the fourteen benches examined in the background laboratory workbench check, only two benches initially showed the presence of any amount of trace explosive. Both benches possessed a white Formica surface, and explosives were recovered from the acetone swab only. Since the background check was qualitative in nature only, the actual amount of explosives recovered was not known. Bench 1 of the background check gives a positive indication for the presence of the explosive RDX. Bench 11 of the background check gives a positive indication for the presence of the explosive TNT.

The five trials from each of the four calibration solutions were averaged and a plot of the calibration solution peak area over the internal standard peak area versus the concentration of the calibration solution over the concentration of the internal standard was created for each calibration solution. The equation that was used to calculate the plot was then used to later determine the amount of explosives recovered from the benchtops in nanograms.

The results obtained from the analysis of the forty explosives samples recovered from the two test workbenches were placed into one of four groups and averaged. The four groups are best described as pre-409 black bench, post-409 black bench, pre-409 white bench, and post-409 white bench. These averages appear below in Table 1.

TABLE 1: Amount of explosives in nanograms deposited and recovered from benches

	EGDN	NG	TNT	PETN	RDX
Theoretical deposition	100	1000	1000	1000	1000
Actual Deposition	127.5	771	996	822	1000
Black bench recovery pre-409®	0	237	106.5	220.5	220.5
Black bench recovery post-409®	0	143.25	55.5	73.5	147.75
White bench recovery pre-409®	0	420	52.5	369.75	502.5
White bench recovery post-409®	0	64.5	21	10.5	84

As the table indicates, the actual amount of explosives recovered is compared to the *theoretical* amount of explosives deposited as well as the *actual* amount of explosives deposited. The theoretical amount of explosives deposited is 1000ng for the explosives NG, TNT, PETN, and RDX, and 100ng for EGDN. The actual amount of explosives deposited was determined by depositing 1mL of the standard explosives solution used in the depositions directly onto a cotton swab and treating the swab in a manner similar to

that of the samples swabbed from the benches. The amount of explosives was then calculated from the chromatograph results using ChromQuest® software.

Upon the completion of the initial swabs of the 40 tiles, each tile was swabbed once again with an acetone swab to determine if any residual explosives could be detected. The swabs were subject to the same treatment as the others, and the results indicated that no explosives remained from either surface.

CONCLUSIONS

It should first be stated that while the initial background check of the fourteen benches used in the examination of explosives evidence in the ATF laboratory did indicate the presence of trace explosives on two of the benches, this check was performed unbeknownst to the scientists and without warning. Many of the scientists had open explosives cases on their benchtops, and as is the general procedure, these benchtops were covered in a protective, wax based paper. The swabbing of the benches took place beneath this paper, an area with which, theoretically, no evidence would ever come in contact. The results of the background check did come as a surprise to some of the scientists; however, no one was concerned that the explosives laboratory had or has an on-going contamination issue.

An initial concern after examining the results of the project itself was the discrepancy between the amount of explosives theoretically deposited and the amount of explosives actually deposited. The most likely explanation is human error. In an experiment of this nature, the possibility for human nature can arise in several forms.

The first possibility for error is in the preparation of the standard solution, but a more likely explanation is loss of explosives due to transfer between the swab and the gloved hand of the scientist performing the recovery, and the extraction procedure itself. Also, the explosives EGDN and NG may succumb to a loss in amount due to evaporation. These reasons may also help to explain why the theoretical amounts of explosives were not recovered from the benches prior to cleaning with 409®.

Based upon the results, it can be determined that more explosives were recovered from the white bench surface than the black bench surface in the case of the trials cleaned with 409® before recovery. The use of 409® also appears to be more effective in the elimination of explosive residues from the white bench surface than the black bench surface. These results can most likely be explained by the nature of each of the surfaces themselves. The black, epoxy resin surface is a more porous, uneven surface than the smooth, white Formica surface. This would account for a smaller amount of explosives being recovered initially, as well as the larger amount of explosives remaining after cleaning the surface with 409® as crevices allow for residues to be trapped and not easily accessible for recovery by swabbing.

The elimination of all traces of explosives from both surfaces in each scenario following a swabbing with an acetone swab indicates that cleaning with 409® is not effective as a sole decontamination technique. Coupling a solvent wipe-down following a cleaning with 409® appears to provide the level of decontamination necessary for the quality of work expected by a forensic science laboratory.

While the ATF laboratory practices careful examination techniques and analysts take precautions when handling evidence, there are no written protocols mandating how

to prepare a work area between cases. Many common procedures are used, but this study helped to validate or invalidate those practices, and recommend a protocol that, when followed, will hopefully control for the possibility of cross-contamination between explosives cases.

Finally, the two bench surfaces analyzed in this project were not only common throughout the ATF laboratory, but throughout many laboratories universally, forensic in nature or otherwise. This study appears to indicate that at least in the area of explosives, the smooth, white Formica workbench surface retained the contaminant explosive solution at a lesser rate than the porous, black epoxy resin surface. This fact may help to promote the use of a similar Formica surface to ensure the maximum level of decontamination possible.

FUTURE RESEARCH

Since the research was carried out in a working laboratory, casework often took precedence over the project. The laboratory possessed only one GC/TEA instrument set-up, and case samples would sometimes occupy the instrument for weeks at a time. Therefore, the initial goals of the project were much more ambitious than what was actually accomplished due to this situation. As is the case for any research endeavor, this project has the possibility for continuation and further exploration in many areas, several of which were included in the initial project proposal.

The first of these areas is the examination of different laboratory surfaces. While the two bench surfaces analyzed in this project are common to many laboratories, other surfaces may be subject to explosives residues, such as floors, instrument surfaces, door

surfaces, and other, less common bench surfaces. A type of bench surface may even exist where a recovery rate close to 100% can be obtained.

Another variable to be explored is the deposition samples. This project looked at explosives residues which can occur in the extraction of a piece of explosives evidence and other scenarios. Another possibility, and one that was initially hoped to be explored by this project, is the deposition of bulk explosives, which are often encountered in explosives casework. The amount and the combination of explosives used can also be varied.

A final variable in this project is the investigation of the effectiveness of other decontamination methods. Possibilities include a further examination of a soap and water method, a bleach solution, solvents other than acetone, and any combination of these methods.

Also, throughout the course of standard explosives evidence analysis, workbench surfaces are covered in a wax-based paper. An interesting aspect of the project that could be explored is the porosity of this paper covering, and whether or not it is durable enough to prevent explosive solutions from seeping through to the bench surface.

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