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A COMPARISON OF THREE TECHNIQUES DEVELOPED FOR SAMPLING AND ANALYSIS OF GUNSHOT RESIDUE BY SCANNING ELECTRON MICROSCOPY AND ENERGY DISPERSIVE X-RAY ANALYSIS

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By

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ABSTRACT

A COMPARISON OF THREE TECHNIQUES DEVELOPED FOR SAMPLING AND ANALYSIS OF GUNSHOT RESIDUE BY SCANNING ELECTRON MICROSCOPY AND ENERGY DISPERSIVE X-RAY ANALYSIS

By

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The purpose of this study was to compare three gunshot residue (GSR) collection methods from hand samples by SEM/EDX analysis. The methods were: the tape lift, glue lift and concentration techniques. Efficiency of particle collection was examined under various conditions including: number of rounds fired, temperature and shelf life. The tape lift surface demonstrated excellent particle collection ability and remained stable for all conditions tested. The glue lift was a relatively inefficient collection surface under all conditions tested. Collection followed by concentration gave highly variable results. An unusually large amount of GSR was found on the hand from working the action of a cleaned weapon. In addition, a survey of U.S. Forensic Crime Laboratories was undertaken to determine methods of GSR analysis. The two general types of GSR analysis were compared and contrasted. Problems encountered by analysts using SEM/EDX were discussed.

DEDICATION

To the little things - "Just because you can't see 'em don't mean they ain't there"

Anonymous drunk, Downtown Detroit, 1973

ACKNOWLEDGEMENT

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INTRODUCTION

A jury's verdict of innocence or guilt in a homicide case may depend on the accuracy and efficiency of gunshot residue collection and analysis. Gunshot residue is a form of transfer or trace evidence. This material can be thought of as loosely complying with the famous Locard exchange principle, derived by the French forensic scientist Edmond Locard in 1910, which states that anytime there is contact between two surfaces, there will be a mutual exchange of matter across the contact boundary. Strictly speaking gunshot residue particles are an example of transfer without direct contact. The reason for collecting and analyzing these particles is obvious and is the basis for the study of trace evidence in forensic science. As was stated quite well by Dr. Paul L. Kirk of the University of California, "It is virtually impossible for a criminal to commit a crime without leaving evidence behind and carrying evidence away with him." (DeForest et al. 1983).

Gunshot residue can be defined as material composed mainly of barium (Ba), antimony (Sb) and lead (Pb) combined to form microscopic spheroid particles, left behind from the primer after discharging a firearm that uses smokeless powder. This material can be collected from the firing hand of a shooter and analyzed by various techniques. The identification of GSR is used frequently in homicide and

suicide cases to demonstrate if the suspect or victim had recently discharged a firearm (Basu et al. 1984). To date, the most definitive method for characterization of gunshot residue (GSR) is by using scanning electron microscopy (SEM) with energy dispersive X-ray analysis (EDX) capability.

The method using SEM-EDX, has several advantages over other current techniques in the analysis of GSR. Unlike neutron activation analysis (NAA) or flameless atomic absorption spectroscopy (FAAS), which are destructive bulk elemental techniques, SEM-EDX is a non-destructive technique which can be applied to a specific particle of GSR. By analyzing distinct particles of GSR, the technique is not subject to spurious results from background or contaminating levels of lead, barium, and antimony because these elements do not combine to form spheroid particles in the environment (Wolten et al. 1979b).

While extensive work has been done demonstrating the ability of SEM-EDX to identify and characterize gunshot residue, there is currently no literature describing how frequently this application of SEM-EDX is being used in Crime Labs throughout the United States. Therefore, a mail survey of Crime Labs across the country was conducted. The first chapter of this thesis addresses the survey results.

The objective of this survey was three-fold:
1) To determine what type of analysis is being performed most frequently on GSR in the laboratory.

2) To establish the average age and dependability of the scanning electron microscopes being used in Crime Labs across the country.

3) To document collection and analysis techniques being used for GSR by SEM-EDX, in the interest of identifying and resolving common problems for the investigator using this technique.

The second chapter of this thesis is a comparison of three techniques used to collect and analyze gunshot residue by SEM/EDX.

While SEM-EDX is conclusive in describing the morphology and elemental composition of the particles in question, it suffers from the excessive time (up to several hours) required to perform the analysis. Several investigators have studied this problem (Basu and Ferriss 1980; Nesbitt et al. 1976; Ward 1982). An ever increasing caseload in the laboratory demands a collection technique that is rapid, dependable and efficient.

Analysis time can be reduced by:

A) Increasing sample concentration. Such a technique was developed by D.C. Ward for concentrating GSR from a Vistanex adhesive coated surface by repetitive centrifugal concentration through a high density liquid.

B) Increasing efficiency of particle lifting from the hand. The tape lift method is said to have excellent particle lifting ability and has been employed by many

researchers (Andrasko and Maehly 1977; Goleb and Midkiff 1975; Matricardi and Kilty 1977; Nesbitt et al. 1976; Tassa et al. 1982b; Wolten et al. 1979b). This technique employs the use of sticky tape (cellotape or 3M Scotch Brand 465) either dabbed against the hand by a police officer wearing rubber gloves or with tape mounted on an aluminum stub (25mm or 5mm in diameter) which is dabbed on the shooter's hand. The stub is sent to the lab where it is coated with a conductive layer of carbon and examined with a scanning electron microscope. An alternative to tape is the glue lift method developed by S. Basu and S. Ferriss (Basu and Ferriss 1980; Basu 1982; Basu et al. 1984). This method uses rubber cement that has been diluted with toluene or 1,1,1,-trichloroethane. A glue layer is applied to a carbon planchet (diameter 1/2") attached to an aluminum pin-type mount for easy handling. Such glue-coated discs are kept in storage boxes until a suspect's hand is to be sampled. The hand is then dabbed three to five times with the sampling disc. The sample is then ready to be examined in the electron microscope, as this method does not require coating the sample with carbon.

C) Decreasing the area to be scanned. Ward scans a 2mm area (Ward 1982), Basu and Ferriss scan several 1.5mm areas (Basu and Ferriss 1980). A general reduction in stub size from 25mm to 5mm has also been employed.

Chapter two of this thesis is a comparative study of

the concentration technique, described by Ward (1982) with modifications by Sugarman (Appendix A); the glue lift technique, described by Basu and Ferriss (1980); and the tape lift technique, as described in the paper by Nesbitt, et al. (1976).

While several methods have been developed for collection of GSR, no thorough investigation has ever been made comparing several of the most common methods for GSR collection and analysis by SEM-EDX. The results of such an investigation should allow crime labs to select a collection technique for GSR based on that technique's relative strengths and weaknesses. Currently, each laboratory must decide somewhat arbitrarily which collection method it will use for GSR analysis by SEM-EDX.

The objectives of this portion of the thesis are to determine which of the three collection techniques tested has:

1) The highest efficiency for lifting particles from the hand.

2) The minimum analysis time.

3) The most stable adhesive surface with respect to temperature and deterioration over time.

Chapter three of this thesis describes the finding of gunshot residue on the hand after working the trigger mechanism of an unloaded, freshly cleaned handgun. Experimental conditions and implications of this observation

are discussed.

LITERATURE REVIEW

The oldest method for determining "gunshot" residue on the hand was introduced in the U.S. in 1933 by Teodoro Gonzales (Cowan and Purdon 1967). It is known by various names including: the Gonzales test, the paraffin test and the dermal nitrate test. It was used to detect nitrates and nitrites left behind on the hand after firing a weapon. A molten wax cast was made of the suspect's hand. This material was then reacted with diphenylamine and diphenylbenzidine which gave pinpoint color reactions with nitrates and nitrites. The technique suffered from several problems including false positives with substances other than nitrates and nitrites, and the presence of nitrates and nitrites on the hands of people who had not fired a weapon. The FBI advised against the use of this test in 1935 (FBI Law Enforcement Bulletin 1935), and again in 1940 (FBI Law Enforcement Bulletin 1940). Despite these warnings, the use of the "paraffin test" continued through the mid 1960's. In 1967, the paraffin test was further discredited in a controlled study which showed no significant difference from paraffin tests done on people that had fired weapons and a "control" group that had not presumably fired a weapon (Cowan and Purdon 1967).

It is interesting to note that Professor Ralph Turner, the former director of the Forensic Science program at MSU

worked on the specificity of the paraffin test as a graduate student (personal communication). He had often wondered if the GSR particles composed of lead, barium and antimony also had nitrates or nitrites associated with their surface, giving rise to the pinpoint color reactions in the paraffin test. In regards to this question, an investigator using Auger electron spectroscopy detected no nitrogen associated with GSR particles (Hellmiss et al. 1987). Those results were corroborated on GSR analyzed using a Cameca Microprobe unit at the University of Michigan, by the author.

Other chemical tests developed to detect gunshot residue were reported in 1959 (Harrison and Gilroy 1959). These methods used sodium rhodizonate for detection of lead and barium and a solution of triphenylmethylarsonium iodide for the detection of antimony. These tests were specific for elements associated with cartridge primers but lacked the sensitivity required to detect small amounts of GSR on the hand. The sodium rhodizonate test is still used quite effectively today to demonstrate lead particles on clothing for firing distance determinations.

Sophisticated instruments capable of detecting microgram quantities of specific elements were employed in an attempt to detect GSR. This type of analysis may be broadly termed "bulk" elemental analysis. A great deal of work was done using neutron activation analysis (NAA) for detection of barium and antimony (Kilty 1975; Kinard and

Lundy 1975; McFarland and McLain 1973; Pillay et al. 1974; Rudzitis and Wahlgren 1975; Rudzitis et al. 1973; Rudzitis 1980). NAA has been used quite frequently in the detection of GSR since the early 1970's. While NAA is a very sensitive technique, it suffers from a variety of problems. Among these are: 1) lengthy analysis and processing times 2) high cost 3) inability to detect lead 4) requires a nuclear reactor 5) interpretation - must know "threshold" values of barium and antimony on the hands of the general population, otherwise results in false positives 6) specificity - high levels of barium and antimony may be from other sources other than GSR and 7) must handle radioactive samples.

In the late 1970's, flameless atomic absorption spectroscopy (FAAS), another type of bulk elemental analysis, was used to detect gunshot residue (Goleb and Midkiff 1975; Kinard and Midkiff 1978; Koons et al. 1988; Newton 1981; Portis and Tilley 1981). The advantage of this technique over NAA was that trace amounts of lead on the hand could also be measured. Additional advantages were shorter processing and analysis times of nonradioactive samples and a relatively low cost of equipment. However, this technique still suffered from the same threshold interpretation and specificity problems as in NAA. A variety of sources exist other than GSR that could cause deposition of elements in question on the hand. Common

sources of lead include (Wolten et al. 1979b): leaded gasoline, old paint and printing inks. Sources of barium are: greases, lubrication oil additives, and extenders for paint and rubber. Sources of antimony include: children's cap guns, and paint (Krishnan 1976).

Other techniques, which have occasionally been used for GSR analysis, include: photoluminescence (Loper et al. 1981), inductively coupled plasma-atomic emission spectroscopy (ICP-AES) (Koons et al. 1988), Auger spectroscopy (Hellmiss 1987), X-ray diffraction (Tassa et al. 1982a) and anodic stripping voltammetry (ASV) (Brihaye et al. 1982).

In the mid 1970's, it was discovered that gunshot residue particles could be visualized by the scanning electron microscope (SEM) (Andrasko and Maehly 1977; Nesbitt et al. 1976; Wolten et al. 1977). GSR particles were found to be generally spheroidal in shape. The use of energy dispersive X-ray analysis allowed the elemental composition of the particles to be determined.

Gunshot residue particles are composed of a variety of elements whose sources are consistent with the primer, bullet, cartridge case and barrel of the gun (Basu 1982; Bergman et al. 1988; Wolten and Nesbitt 1980). An extensive report was made by the Aerospace Corporation (Wolten et al. 1977), that described, in essentially a tutorial fashion, how to optimize the operational variables of the SEM in

order to obtain a good backscatter image of GSR particles. The report goes on to examine various types of ammunition used and weapons fired in an attempt to characterize the type of GSR found in test firings.

Other researchers investigated the formation of GSR and the location of the elements associated with GSR in the spheroid particles (Basu 1982; Burnett 1989; Tassa and Zeldes 1979; Tassa et al. 1982a). The spheroidal shape of GSR is assumed to be caused by the condensation of elements in the primer from a molten state when a gun is fired. In general, particles of ten microns or larger are composed mainly of barium. In the smaller particles, the elements lead, barium and antimony are mixed throughout the particle unless the particle was nodulated. In the case of a particle with nodules, the nodules are typically composed of lead and sometimes antimony. The body of the particle is composed of barium with occasional antimony present. The data were obtained by X-ray mapping individual particles either whole or in cross section.

The major advantage of using SEM/EDX to analyze GSR is the ability to locate single particles with unique elemental composition (Ba, Pb, and Sb) indicative of GSR. Studies were made that demonstrated the unique elemental composition of GSR compared to particles in the environment or on the hands of people in occupations that could potentially produce particles similar to GSR (Nesbitt et al. 1976;

Wolten et al. 1979b).

A great deal of the literature on the analysis of GSR by SEM/EDX describes various types of ammunition used and weapons fired to determine the amount and type of GSR produced (Andrasko and Maehly 1977; Matricardi and Kilty 1977; Taylor et al. 1979; Wolten et al. 1977; Wolten et al. 1979a). Ideally, one would like to be able to match the type of ammunition used to the type of GSR particles found on the hand of a shooter. Unfortunately, this is not usually possible, at least with ammunition found in the U.S.

There have been a number of papers published describing the use of SEM/EDX in analysis of GSR in actual casework (Basu et al. 1984; Krishnan 1976; Bergman et al. 1988). While in most instances the type of GSR left on the hand can not be associated with the type of ammunition used, particles of the bullet left behind along the "wound track" can sometimes be used to identify the type of ammunition used and, thereby, the particular weapon used (Taylor et al. 1979).

Over time a variety of collection and/or concentration techniques have been developed for analysis of GSR by SEM/EDX (Andrasko and Maehly 1977; Basu and Ferriss 1980; Gansau and Becker 1982; Matricardi and Kilty 1977; Portis and Tilley 1981; Sild and Pausak 1979; Tassa et al. 1982b; Wallace and Keeley 1979; Ward 1982). There are three general types of collection devices: 1) tape lift

(Andrasko and Maehly 1977; Gansau and Becker 1982; Matricardi and Kilty 1977; Tassa et al. 1982b) 2) glue lift (Basu and Ferriss 1984) and 3) concentration (Portis and Tilley 1981; Sild and Pausak 1979; Wallace and Keeley 1979; Ward 1982).

Among the tape lift techniques, a variety of different types of tape have been tested (Andrasko and Maehly 1977). The type of tape lift cited most often in the literature is 3M type 465 adhesive transfer tape.

The glue lift collector consists of a thin layer of diluted rubber cement on a polished carbon planchet.

Concentration methods vary; one employs suction of particles directly onto an adhesive coated disc (Sild and Pausak 1979), another allows examination of GSR by both FAA and SEM/EDX (Portis and Tilley 1981). In this case, the sample is collected from the hand using isopropanol swabs and then concentrated onto a filter using a 5 ml syringe. The other two concentration methods mentioned used a diluted Vistanex (glue) coated stub to lift the particles off the hand. Particles were then dissolved out of the Vistanex and passed through the concentration device. In the one case, concentration was achieved by suction filtration (Wallace and Keeley 1979); in the other case by centrifugation (Ward 1982). The centrifugation procedure also allowed for addition of high density solvents to float off contaminating debris (Epidermal cells, hair, fibers etc.).

A major disadvantage of SEM/EDX analysis is lengthy analysis time and operator fatigue. In an effort to overcome this problem, various automated GSR analysis systems for SEM/EDX systems have been developed (DeForest et al. 1983; Tillman 1987; White and Owens 1987). Currently, there are a number of energy dispersive X-ray systems on the market that are designed to handle automated particle search and recognition of GSR. The manufacturers include: Link Analytical Systems, Tracor Northern and Kevex.

CHAPTER ONE

SURVEY OF GUNSHOT RESIDUE ANALYSIS IN FORENSIC SCIENCE LABORATORIES ACROSS THE UNITED STATES.

INTRODUCTION

There are two general types of methods currently used for analysis of gunshot residue from the hands of a shooter. One type, may be termed "Bulk Elemental Analysis Techniques", which includes: flameless atomic absorption (FAA) (Koons et al. 1989), neutron activation analysis (NAA) (Hoffman 1975), inductively coupled plasma-atomic emission spectrometry (ICP-AES) (Koons et al. 1989), and anodic stripping voltammetry (ASV) (Brihaye et al. 1982). The other common type of GSR analysis is by scanning electron microscopy with energy dispersive X-ray capability (SEM/EDX) (Wolten et al. 1977).

Gunshot residue is encountered frequently as evidence in homicide and suicide cases. However, not all forensic science laboratories choose to analyze this evidence. Those that do, have a variety of analysis methods to choose from. To determine who is analyzing GSR and by what means, a nationwide survey of forensic science laboratories was undertaken.

The purpose of this survey was three-fold:

1) To determine the methods of analysis being used nationwide on gunshot residue samples in forensic science laboratories.

2) To compare and contrast the two general types of methods being used to analyze GSR.

3) To document the procedures and types of equipment being used in GSR analysis by SEM/EDX in the interest of identifying and resolving common problems for the investigators using these techniques.

MATERIALS AND METHODS

The survey was mailed to two hundred Forensic Science Labs in every state in the U.S.. The data were then collected and tabulated. A report summarizing the data and conclusions drawn from the information collected was available upon request to all participating laboratories. A cover letter, to help elicit cooperation by the Labs, was also sent with this survey. Results of the survey were submitted for publication to the Journal of Forensic Sciences in July, 1989.

DESCRIPTION OF SURVEY INSTRUMENT

A mail survey was conducted on two hundred forensic science laboratories distributed to all fifty states in the U.S. in November of 1988. The response rate to the first mailing of the survey was 51.0%. A second mailing of the survey in December of 1988 brought the response rate up to 71.5%. A copy of the survey instrument appears as Appendix C.

RESULTS AND DISCUSSION

Table 1 lists the percentage of laboratories analyzing gunshot residue and the method used for GSR analysis. A total of 57% of the labs responding don't analyze gunshot residue themselves; 52% of those labs send GSR samples either to the FBI or a state/regional lab for analysis. Of the labs analyzing GSR, 57% use a bulk elemental analysis technique, 34% employ SEM/EDX alone or combined with FAA. It is of interest to note that while X-ray Fluorescence and Photoluminescence have been used in the past to analyze GSR, no labs indicated the use of those techniques in this survey.

The results of the survey indicate that thirty-one states have at least one lab conducting GSR by one of the methods listed above. Fourteen states had no labs conducting GSR analysis by the above methods. In five states, no labs responded.

Some interesting results are obtained when comparing GSR analysis techniques. One of the main contentions for using bulk elemental analysis techniques over SEM/EDX in the past has been the shorter analysis times of the former technique. The survey data indicate however, that on the average, the amount of time spent per analysis using either technique is about the same. In fact, the mean time required to analyze a sample, as well as the mean time spent

Table 1. Percent of forensic laboratories analyzing gunshot residue and method used for analysis.

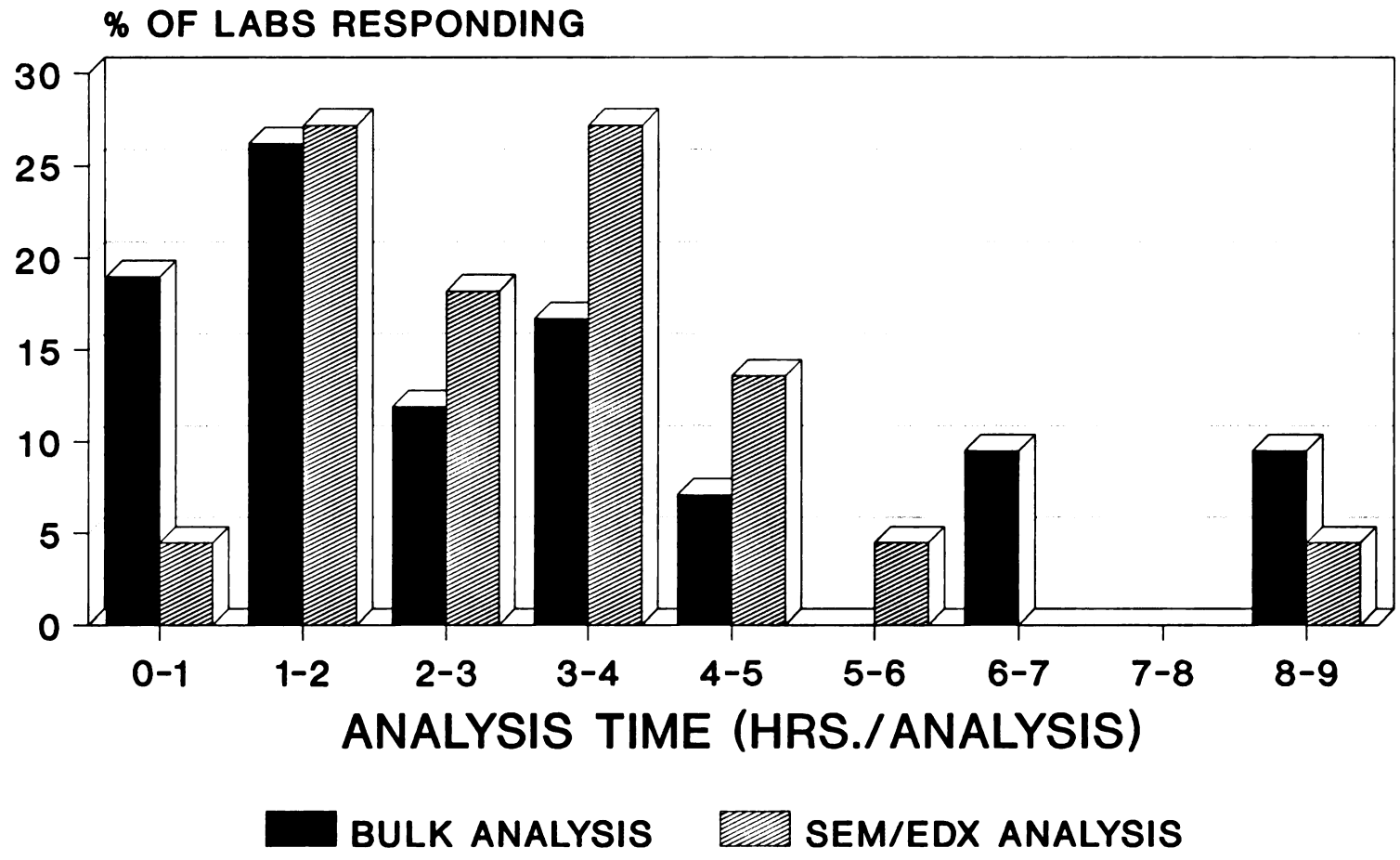
	% of Labs Responding	N
Where Analyzed		
GSR Analyzed in Lab	43%	144
Sent to FBI	15.3%	
Sent to State/Regional Lab	14.6%	
Not Done	27.1%	
Method of GSR Analysis		
FAA	48.4%	62
SEM/EDX Alone	21.0%	
SEM/EDX Combined with FAA	12.9%	
NAA	1.6%	
ASV	4.8%	
Microchemical Tests	11.2%	

on GSR analysis per week, was fairly similar for both bulk analysis and SEM/EDX techniques: 3.0 hrs per analysis vs 3.1 hrs per analysis, and 13.3 hrs per week vs 15.9 hrs per week, respectively (Figures 1 and 2).

Survey participants were asked to respond to the question of how frequently and on what grounds GSR analysis was challenged in court. The response rate for this particular question was relatively low but still bears examination. Table 2 lists how frequently GSR analysis is challenged in court and Table 3 lists the grounds for challenge. Bulk analysis techniques are challenged slightly more often, mainly on the grounds of specificity. This may be a significant concern with respect to the potential for false positives. SEM/EDX analysis, when challenged, is usually challenged on the examiner's interpretation of the data. Since there are well defined, accepted, characteristic criteria for defining gunshot residue by this technique, it is less likely to lead to false positives.

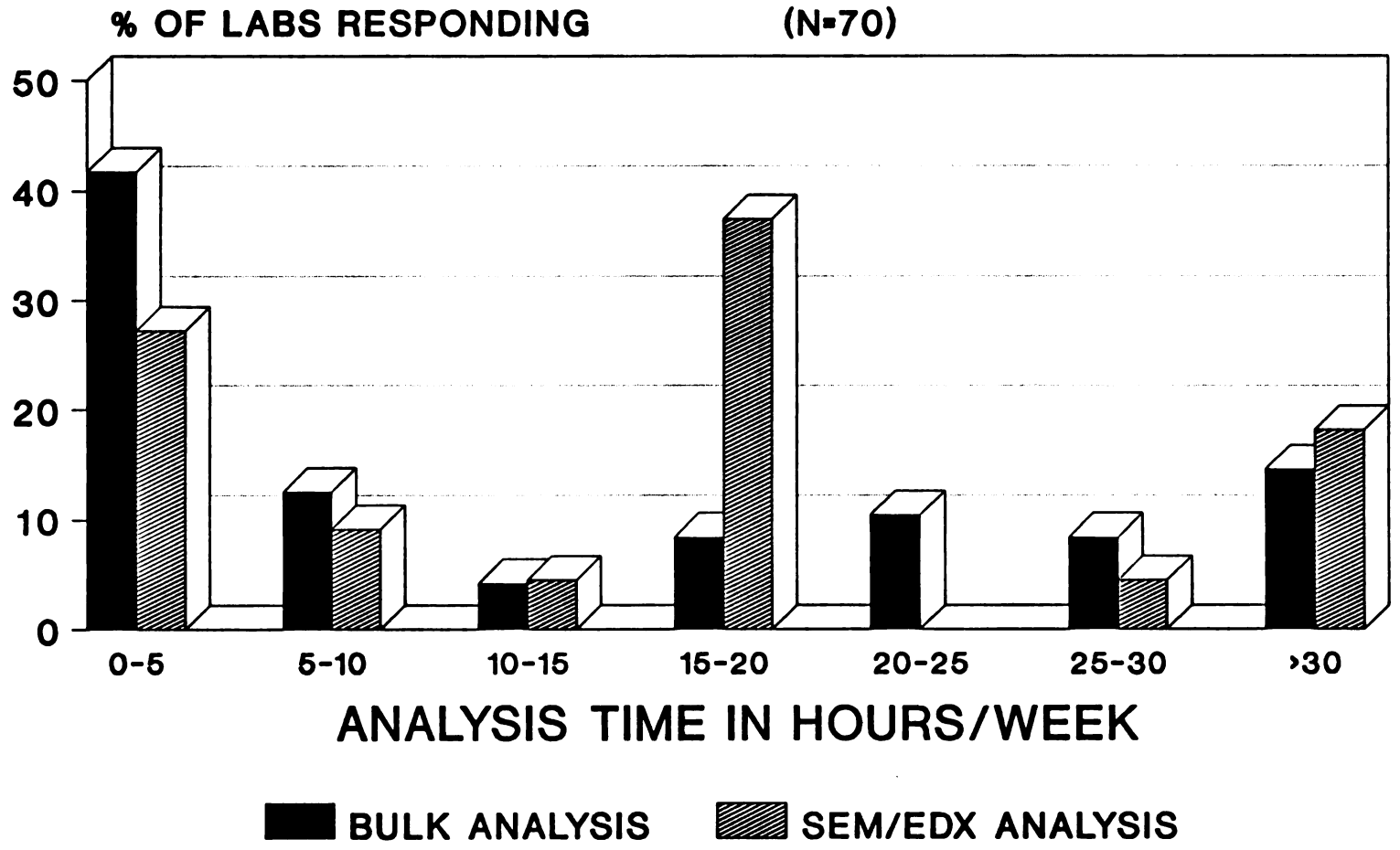
Currently, 54% of the labs with SEM/EDX capability use their instrument for GSR analysis. Table 4 lists the type of SEM equipment being used by forensic science labs and its age and dependability. The scanning electron microscopes used most frequently throughout the country are listed in Table 4. Tracor Northern was the most frequently used EDX system (35%), with EDAX following with 27%, Princeton Gamma Tech 24% and Kevex with 15%, (n=34). The mean age of

Figure 1. Time required per analysis for labs analyzing gunshot residue.



1 BULK ANALYSIS (N=42) MEAN=3.02 S.D.=2.29
SEM/EDX (N=22) MEAN=3.09 S.D.=1.72

Figure 2. Analysis time in hours per week for labs conducting GSR analysis.



2 BULK ANALYSIS- MEAN=13.3 S.D.=11.6
SEM/EDX ANALYSIS- MEAN=15.9 S.D.=10.6

Table 2. Frequency of challenge to GSR analysis in court.

	Frequency of Challenge (Number of Labs Responding)				
	0%	Rarely (1-10%)	10-30%	>50%	N
Method of Analysis					
Bulk Analysis[1]	10	12	5	2	29
SEM/EDX Alone	3	5	1		9
SEM/EDX with FAA	2	4			6

[1]Includes-Flameless atomic absorption, neutron activation, inductively coupled plasma-atomic emission, and anodic stripping voltammetry

Table 3. Grounds for challenge in court.

	METHOD OF ANALYSIS (# of labs responding)		
	Bulk Analysis	SEM/EDX Alone	SEM/EDX & FAA Combined
<u>Basis for challenge</u>			
Not Specific for GSR	10	1	1(FAA)
Did Defendant Fire a Gun?	4		
Interpretation of Threshold Level	4		
Interpretation in General		4	3
Evidence Consumed	2		
Operator Proficiency		1	
EDX Sensitivity		1	
Collection Technique		1	

Table 4. Laboratory SEM equipment, age and dependability.

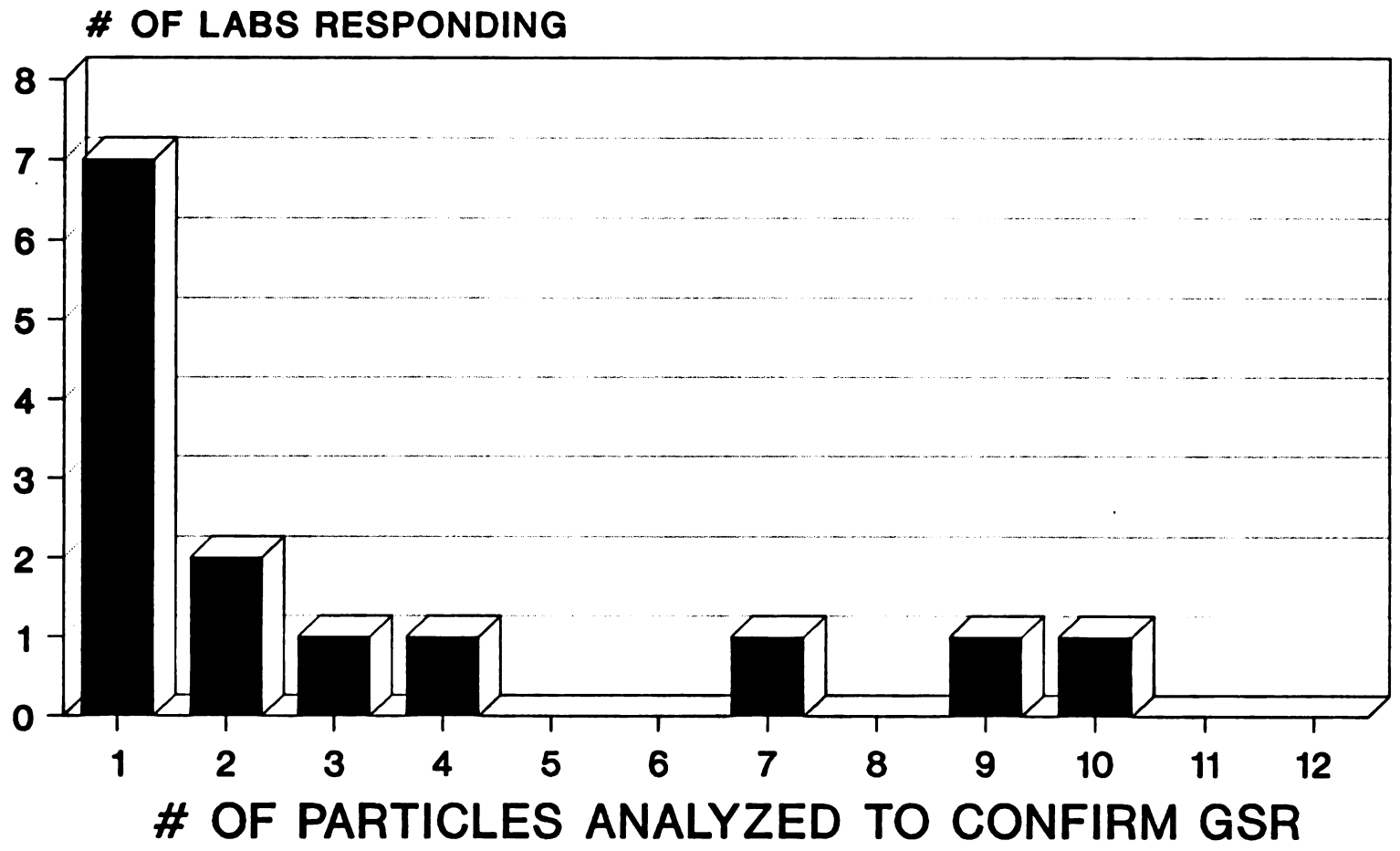
	% of Labs Responding	Mean	Standard Deviation	N
Make of SEM Used				
ISI	28.6%			35
AMRAY	22.9%			
CAMBRIDGE	14.3%			
HITACHI	11.4%			
CAMSCAN	8.6%			
ETEC	5.7%			
JEOL	5.7%			
BAUSCH & LOMB	2.9%			
Make of X-Ray Analyzer				
TRACOR NORTHERN	35.3%			34
EDAX	26.5%			
PRINCETON GAMMA TECH	23.5%			
KEVEX	14.7%			
Age of SEM				
1-5yrs.	55.6%	6.57yrs.	4.77yrs.	36
6-10yrs.	30.6%			
11-15yrs.	8.3%	(5.0yrs.=Median)		
16-20yrs.	5.6%			
# of Weeks SEM/EDX was Down				
0-1	26.7%	2.8wks.	6.8wks.	30
1-2	26.7%			
2-3	24.3%	(1.5wks.=Median)		
3-4	13.3%			
4-5	6.7%			
40 wks.	3.3%			

scanning electron microscopes being used was 6.57 years with a standard deviation of 4.77 years and a range of 1 to 20 years old, (n=36). As an estimate of instrument dependability, the mean number of weeks the SEM/EDX system was "down" in 1988 was 2.81 weeks with a standard deviation of 6.81 weeks and a range of 0 to 40 weeks, (n=30). As far as the type of collection technique being used most frequently by labs analyzing GSR by SEM/EDX, a tape lift technique was used by 48% of the labs responding; followed by a concentration technique (16%), glue lift (12%), swab for FAA & SEM/EDX, (12%), vacuum suction (4%) and other techniques (8%), (n=25).

A potential problem that exists in using SEM/EDX for GSR analysis is the variation between labs in determining the minimum number of particles analyzed to confirm gunshot residue (Figure 3). A total of 41% of the labs responding, reported that finding one particle which meets the shape and elemental characteristics of GSR is enough to confirm GSR on the hand (n=17). However, the range of responses to this question ran from 1 to 10 particles. Some responses gave options such as, "1-2 unique" or "8-10 characteristic particles", " depends on the type of particle", or "none set; based on particles & FAA". What should be the accepted standard is an important question which needs attention by experts in the field of GSR analysis using SEM/EDX.

Other problems cited by laboratories using GSR analysis

Figure 3. Variations in the minimum number of particles analyzed by SEM/EDX to confirm GSR.



OTHER: 1-2 UNIQUE OR 8-10 CHARACTERISTIC
DEPENDS ON TYPE OF PARTICLE
NONE SET BASED ON PARTICLES & FAA

by SEM/EDX were as follows:

1) Glue lift efficiency drops with wet, bloody or dirty hands.

A possible solution to this problem would be to use a stickier medium such as 3M adhesive transfer tape; although dirty hands will remain a potential problem.

2) Nucleopore filter tends to clog in the concentration technique. This observation is in agreement with the findings of Zeichner et al. (1989). Their recommendation was to eliminate the concentration step and observe the glue or tape lift directly. Dennis Ward at the FBI laboratory in Washington D.C., as well as Loren Sugarman at the Orange County Sheriff-Coroner Department, Santa Ana, CA. suggest the centrifugal force used in concentration can be critical. Too high of a g force may pellet debris on the filter (personal communication).

3) Lengthy analysis times, as well as the analysis being fatiguing to the operator, especially on negative samples. An observation was also made that it is difficult to find a method conducive to both SEM/EDX and FAA. A possible solution to this problem, which is currently being used in some labs, is to collect samples for SEM/EDX from the web area of the hand and possibly the face. In addition, swabs from the back and palm of the hands are collected. FAA is then used as a screening technique and only potential positive samples are analyzed by SEM/EDX.

Dr. Robin Keeley from the Metropolitan Police Forensic Science Laboratory (Scotland Yard) points out however, that FAA is a relatively insensitive technique with respect to GSR. One may actually have over a hundred particles of GSR (assuming an average particle size of 3 micrometers) and still fall below the threshold level for Pb, Ba and Sb as detected by FAA (personal communication).

4) GSR is collected too long after the incident occurs. This is an inherent problem. Stressing the need for collecting samples as soon as possible would be helpful.

5) Cigarette lighter flint particles mimic GSR in morphology and increase analysis time. This is an interesting observation for which there is no proposed solution at this time.

SUMMARY AND CONCLUSIONS

To determine who is analyzing GSR and by what means, a nationwide survey of forensic science laboratories was undertaken. Data on gunshot residue analysis were obtained from a mail survey of two hundred forensic laboratories in the U.S. with a response rate of 71.5%. Over half of the labs responding don't analyze gunshot residue themselves; 52% of those labs, send GSR samples either to the FBI or a regional lab for analysis. Of the labs analyzing GSR, 57% use a bulk elemental analysis technique, 34% employ SEM/EDX alone or combined with FAA. Interestingly, the mean time required to analyze a sample, as well as the mean time spent on GSR analysis/week, was fairly similar for both bulk analysis and SEM/EDX techniques. Currently, about half of the labs with a SEM/EDX use it for GSR analysis. Bulk analysis techniques are challenged slightly more often in court, mainly on the grounds of specificity. This may be a significant concern, with respect to the potential of reporting false positives. A number of problems and potential solutions encountered by investigators using the SEM/EDX technique for GSR analysis were discussed. With the commercial availability of automated gunshot residue programs for SEM/EDX equipment a shift towards this type of analysis may appear in the future.

CHAPTER TWO

A COMPARISON OF THREE TECHNIQUES DEVELOPED FOR SAMPLING AND ANALYSIS OF GSR BY SEM/EDX ANALYSIS

INTRODUCTION

The objectives of this portion of the thesis are to determine which of the three collection techniques tested has:

1) The highest efficiency for lifting particles from the hand.

2) The minimum analysis time.

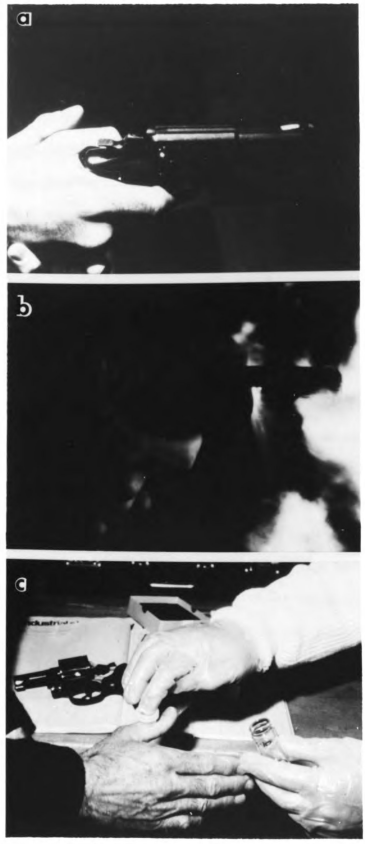
3) The most stable adhesive surface with respect to temperature and deterioration over time.

MATERIALS AND METHODS

Ammunition - Federal .38 special, 125 grain, jacketed soft point, Lot# 12A. Weapon - Smith and Wesson .38 special caliber, model 10-8, 6 shot revolver with a two and one half inch barrel. Electron Microscope - JEOL 35C SEM with a solid state backscatter detector and equipped with a Tracor Northern 5500 energy dispersive X-ray analyzer (EDX). Carbon Coating - Ladd Vacuum Evaporator, Ladd Research Industries Inc., P.O. Box 1005, Burlington, VT 05402. Carbon Planchets - Specially smoothed surface, carbon specimen mounts (disc thickness 5mm, diam. 15mm), Ladd Research Industries Inc., P.O. Box 1005, Burlington, VT 05402. Gun Cleaning Solvent - Brite Bore, Mill Run Products CO., Cleveland, OH 44104. Gun Cleaning Patches - Hoppe's #1204 .38 - .45 cal., Airport Industrial Mall, Coatsville, PA 19320. Concentration Devices - See Appendix A for materials and methods according to Sugarman. Centrifuge - Precision Vari-Hi-Speed Centricone, Precision Scientific Company, Chicago, IL 60647.

Firing and Collection - (Figure 4) The shooter washed and dried the hands prior to firing. The firing distance was 32 inches from barrel to target face ("cotton box"). The shooter used a weapon that had been cleaned prior to firing. The same weapon was used for all firings. After

Figure 4. Gunshot residue collection from the hands of a shooter. 4a) Shooter holding Smith & Wesson model 10-8 prior to firing. 4b) Muzzle blast from firing one round of Federal .38 special ammunition. 4c) Firing hand sampled with a collection device used in the concentration technique.



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firing, the shooter left the "firing room" and his hand was sampled by the researcher. The researcher wore latex gloves and sampled the thumb, web and index finger of the firing hand by successive dabbings of the hand with the collection stub (Figure 4c). The collection stub was then numbered and covered with its protective cap. The collection stub was removed from its support piece (rubber stopper) and carbon-coated in the vacuum evaporator or processed through the concentration technique (Appendices A & B).

Hand and gun controls - Before analysis on the electron microscope, all samples were given a random number using a table of random numbers. The analyst knew what type of collection device was being analyzed by the surface characteristics of the device but had no idea what treatment the collection device had received or whether it was a control collector. For every treatment, five repetitions were made and two controls were run. The controls consisted of a "hand control" where the subject who had been firing the revolver washed his hands and was then sampled, and a "gun control" where the revolver was cleaned immediately after firing by running a solvent (Brite bore) soaked patch on a .38 caliber copper brush down the barrel and cylinder chambers several times. This procedure was repeated with a second solvent soaked patch which was also used to clean off debris on all metal surfaces of the revolver with the cylinder removed (open). A third "dry" patch was then run

through the bore and chambers as well as cleaning all metal surfaces as described above. The revolver grips and exterior were then blown off with compressed air. At this point, the shooter washed his hands and picked up the cleaned revolver working the action three times by pulling the trigger on empty chambers. The hand was immediately sampled.

Collection devices - Tape lift and glue lift collection devices consisted of a number 3 inverted stopper that had a 15mm diameter aluminum stub mounted on top of it using 3M brand 465 adhesive transfer tape. The surface of the aluminum stub was coated with one of the following adhesive surfaces to be tested:

1) 3M brand 465 adhesive transfer tape (Nesbitt et al. 1979).

2) 3M brand 465 adhesive transfer tape fixed to a 15mm diameter polished carbon planchet coated with rubber cement diluted 1:4 with toluene (Basu and Ferriss 1980; Basu 1982; Basu et al. 1984).

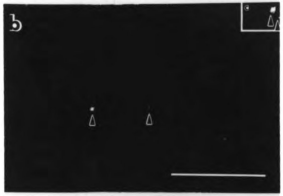
Each collection device was covered by a protective cap consisting of a number 20 plastic test tube closure. Devices were stored at room temperature and used within 24 hours after preparation, unless otherwise noted.

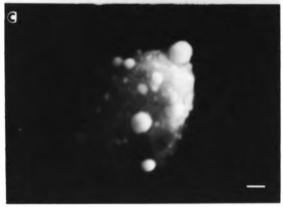
Collection devices for the concentration technique consisted of 3M brand double stick tape bound to a 15mm diameter mylar surface that is coated with Vistanex adhesive

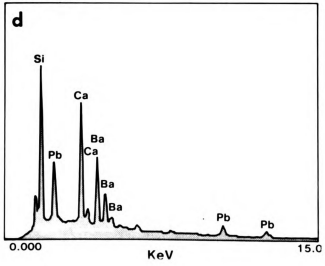
diluted to 15% with hexane (Appendix A) (Figure 4c).

Electron Microscopy - The JEOL 35C was operated at an accelerating voltage of 25kV. The lithium-drifted silicon crystal of the X-ray detector was kept in liquid nitrogen at a distance of 55mm from the center of the column. The working distance for the specimen was 39mm. A brass specimen holder with a 25mm diameter was lined with an aluminum adapter to accommodate the 15mm diameter stub being analyzed. An objective aperture setting of 600 micrometers was used to obtain increased signal for backscatter detection. A known sample of gunshot residue collected on 3M brand 465 adhesive transfer tape (3 rounds fired) was inserted into the microscope as a standard for fine adjustment of the backscatter image (Figure 5). The image was focused at 300X in the secondary electron image (SEI) mode. The backscatter image, which is sensitive to increasing atomic number, was collected using the slow scanning option while adjusting the gain and contrast to give a dark background with GSR appearing as bright circular white spots with a circular halo around them. A particle was selected and the magnification was increased so that the particle image filled the majority of the screen. The image was refocused in the SEI mode and a X-ray spectrum was accumulated for 70 seconds at a beam current of 550 picoamps. The particle was confirmed as gunshot residue if it fell into one of the following four categories:

Figure 5. Location and identification of GSR by SEM/EDX. 5a) Secondary electron image of a tape lift surface used to collect GSR from hand after firing three rounds of Remington .38 special ammunition. Arrowheads point to potential gunshot residue particles. Magnification - 30X, bar = 1000 micrometers. 5b) Backscatter electron image of the same field as 5a. Arrowheads point to potential GSR particles. Large particle in inset is enlarged in micrograph 5c. Magnification - 30X, bar = 1000 micrometers. 5c) Secondary electron image of large nodulated GSR particle from inset in 5b. Magnification - 5500X, bar = 1 micrometer. 5d) Energy dispersive X-ray spectrum from GSR particle in 5c.







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1) Lead (Pb), Barium (Ba), and Antimony (Sb).

2) Ba and Sb.

3) Ba, Calcium (Ca), Silicon (Si) with a trace of Sulfur (S).

4) Ba, Ca, Si with a trace of Pb provided that no Zinc (Zn) was present (residue from stud guns has been found to contain Ba, Ca, Si, Pb, Cu and Zn) (Wolten et al. 1979b).

OPERATIONALIZING THE VARIABLES

Dependent Variables

1) Efficiency of collection - Efficiency of collection is defined as: The number of GSR particles found in one hour searching at 300X magnification in the backscatter mode in the SEM. If five GSR particles were found in less than one hour, the time taken to find the particles was recorded and the number of particles found in one hour was extrapolated according the equation:

60 minutes/search time (minutes) X # of particles found = number particles found per hour

This analysis assumes that GSR was distributed randomly on the stub (Wolten et al. 1977).

2) Analysis time - Analysis time is defined as the time required from insertion of the sample into the electron microscope (time zero) to the moment that five particles

whose spectra and morphology characterize them as GSR, have been found and saved to disk. An upper limit of one hour was set for the analysis time per sample.

3) Concentration time - This refers only to the collection technique using the Vistanex adhesive. Concentration time is defined as: the time from removal of the mylar surface to the moment that the 0.45 um filter was dry and ready to be carbon coated.

Independent Variables

1) Number of rounds fired - Either one or three rounds were fired. Sampling was conducted using a collection device prepared 24 hours in advance and stored at room temperature.

2) Temperature - Holding the number of rounds fired constant at three, each collection device was prepared 24 hours in advance keeping the devices at a temperature of 56 degrees Celsius or -4 degrees Celsius for 12 hours prior to sampling.

3) Time - Holding the number of rounds fired constant at three, and the temperature constant at 22 degrees Centigrade (room temperature) collection devices were prepared and stored for three weeks or six weeks prior to sampling.

STATISTICS

Three different methods of collecting GSR were examined. They were: 1) The tape lift 2) The glue lift 3) A Vistanex glue lift followed by concentration via centrifugation.

For each collection method six different treatments were examined. The treatments were: 1) three rounds fired 2) one round fired 3) collection device stored twelve hours at -4 degrees Centigrade 4) collection devices stored twelve hours at 56 degrees Centigrade 5) collection devices stored for three weeks prior to use 6) collection devices stored for six weeks prior to use. Unless otherwise stated, collection devices were prepared 24 hours in advance and residue was collected from three rounds being fired.

For each treatment there were five repetitions. The gun was cleaned between each repetition and both the shooter and the investigator collecting GSR washed their hands between repetitions.

In addition to the five repetitions for each treatment a "hand control" and a "gun control" sample was collected from the hand (see Materials and Methods section for firing and hand sampling procedure). Washing of the hands is usually sufficient to remove gunshot residue, however, depending on how thoroughly the hands are washed, some GSR may remain (Harrison and Gilroy 1959).

Unfortunately, the data from each method were not

collected in a totally random fashion. Reasons for this were two-fold. 1) The concentration method required a special size 0.45 micron Nucleopore filter, which had to be special ordered. This item was backordered for several months, at which point it was decided to begin collecting GSR using the tape lift method. 2) The treatments of collectors prepared three and six weeks in advance made a totally random design impractical.

GSR collections were made one method at a time beginning with the tape lift method. A single treatment consisting of five repetitions and a "hand " and "gun" control was completed on a given day. After all GSR collections were made for the tape lift method and the glue lift method, the collection devices were renumbered using a table of random numbers and analyzed in a totally random fashion on the electron microscope. When the 0.45 micron filters arrived for the Vistanex glue lift concentration method, samples were collected and random numbers assigned for analysis as described above.

Since the experimental design was not random between methods, the significance of statistical analysis between methods should be interpreted with caution. However, within a given method, the collection devices were analyzed in an independent and random fashion allowing an analysis of variance to be made between treatments within a single method.

Assuming a random distribution of particles on the collection surface (Wolten et al. 1977), particle count data were normalized to time. For ease of statistical manipulation, the number of particles per one hour search time was determined.

Search time was equal to the time taken to find five GSR particles, or 60 minutes if less than five GSR particles were found.

The ln (x+1) transformation was required to fulfill the homogeneity of variance assumption of ANOVA. Homogeneity of variance within method and between treatments was examined with Bartlett's test (Steel and Torrie 1980). Means from ln (x+1) transformed data were separated via the SNK (Student Newman Keuls) procedure or Student's t test following ANOVA (Proc. GLM, SAS, 1983) (SAS Institute Inc.).

RESULTS

COMPARISON BETWEEN COLLECTION METHODS

An indication of the collection device particle lifting efficiency was reflected by the mean number of particles found per hour. Obviously, the more particles found per unit time the greater the efficiency of collection. The data in Table 5 indicated that the ln(x+1) particle per hour means were significantly different from each other at the .05 alpha level for the three collection methods tested.

If one examines the particles/hr. mean for the tape lift method the mean particles/hr. was greater than five. In other words, on the average, it took less than one hour to find five particles of GSR on the tape lift surfaces (Table 5).

The particle per hour means for both the concentration technique and the glue lift were much lower than in the tape lift method. However, since the experimental design was not random, comparisons between methods must be considered carefully.

COMPARISONS WITHIN COLLECTION METHOD

The data in Table 6 suggest that the collection efficiency of the 3M type 465 adhesive transfer tape was stable under all conditions tested. The SNK test showed a

Table 5. Statistical analysis of the ln (x+1) of particle per hour means between collection methods.[1]

Method	SNK[2] Grouping	Mean (ln (x+1) of particles per hour)	N	Mean (particles per hour)
Tape lift	A	1.973	30	6.192
Concentration technique	B	1.081	30	1.948
Glue lift	C	0.538	30	0.713

[1] Calculated by SNK procedure using analysis of variance.

[2] Means sharing the same letter are not significantly different from each other at alpha = .05 level.

Table 6. Statistical analysis[1] of the ln (x+1) of particle per hour means for tape lifts.

Treatment	SNK[2] Grouping	Mean (ln (x+1) of particles per hour)	N	Mean (particles per hour)
1 Round fired	A	2.233	5	9.380
3 Rounds fired	A	2.188	5	8.100
-4 Degrees C.	A	2.105	5	8.320
3 Weeks old	A	1.938	5	7.540
Gun control	A	1.705	5	5.517
56 Degrees C.	A	1.694	5	5.240
6 Weeks old	A	1.678	5	6.128
Hand control	B	0.384	5	2.567

[1] Calculated by SNK procedure using general linear models.

[2] Means sharing the same letter are not significantly different from each other at alpha = .05 level.

significant difference in means (ln (x+1) of particles/hr.) at an alpha level of .05 for the hand control data compared to the other treatments, as would be expected. A result which was not expected was that the gun control data showed no significant difference in mean (ln (x+1) of particles/hr.) when compared to the other treatments. This observation will be discussed in detail later in chapter 3.

The concentration technique gave highly variable results between treatments tested. A comparison of the ln (x+1) of particle/hr. means for the treatments in the concentration method by SNK, resulted in means which were significantly different at the .05 alpha level (Table 7). Namely, one round fired samples had the highest mean and was significantly different from the six week old collections, gun controls and three rounds fired. The potential source of the decreased particle counts found in the concentration method will be examined in the discussion section later in this chapter.

In the present study, statistical analysis by SNK of ln (x+1) of particles/hr. means for the glue lift method showed no significant differences between hand controls and any of the treatments examined (Table 8). Too few particles per stub surface were found. This is indicative of an inefficient particle lifting surface. Note the mean number of particles found per hour for the glue lift technique in Table 8.

Table 7. Statistical analysis of the $\ln_1 (x+1)$ of particles per hour means for concentration method.[1]

Treatment	SNK[2] Grouping		Mean (ln (x+1) of particles per hour)	N	Mean (particles per hour)
1 Round fired	A		1.828	5	6.080
3 Weeks old	A	B	1.520	5	4.160
56 Degrees C	A	B	1.430	5	4.320
Hand control	A	B	0.855	5	1.600
-4 Degrees C	A	B	0.748	5	1.800
6 Weeks old		B	0.599	5	1.200
Gun control		B	0.462	5	0.833
3 Rounds fired		B	0.358	5	0.600

[1] Calculated by SNK procedure using general linear models.

[2] Means sharing the same letter are not significantly different from each other at alpha = .05 level.

Table 8. Statistical analysis[1] of the ln (x+1) of particles per hour means for glue lifts.

Treatment	SNK[2] Grouping	Mean (ln (x+1) of particles per hour)	N	Mean (particles per hour)
1 Round fired	A	1.011	5	2.540
3 Rounds fired	A	0.832	5	1.800
3 Weeks old	A	0.555	5	0.800
6 Weeks old	A	0.416	5	0.600
Gun control	A	0.366	5	0.667
56 Degrees C	A	0.277	5	0.400
-4 Degrees C	A	0.139	5	0.200
Hand control	A	0.000	5	0.000

[1] Calculated by SNK procedure using general linear models.

[2] Means sharing the same letter are not significantly different from each other at alpha = .05 level.

In order to test the efficiency of the glue lift technique, an experiment was designed as described in the procedure for three rounds fired, where the hand was sampled by twelve dabs with the glue lift, the same area of the hand was then sampled with a tape lift collector. The procedure was repeated five times with gun cleaning and hand sampling as described previously. Tape lift stubs were carbon coated and all stubs were assigned random numbers and analyzed on the electron microscope.

An ANOVA by Student's t test indicated a significant difference in the ln (x+1) of particle/hr. means for the glue lift and tape lift collectors, alpha = .05. These results demonstrated the less efficient particle lifting surface of the glue lift devices (Table 9). Decreased collection efficiency was reflected not only by the particle/hr. means being much lower in the glue lift vs the tape lift, but also in the fact that particles collected on the tape lift surface represent GSR, which was left on the hand after initially sampling the hand with a glue lift device.

Table 9. Statistical analysis of ln (x+1) of particles per hour means for glue lifts followed by tape lifts.[1]

Method	T^2 Grouping	Mean (ln (x+1) of particles per hour)	N	Mean (particles per hour)
Tape lift	A	1.788	5	6.040
Glue lift	B	0.8832	5	1.400

[1] Calculated by Students t test (LSD).

[2] Means sharing the same letter are not significantly different from each other at alpha = .05 level.

DISCUSSION

The tape lift collection devices in this study proved to be the most efficient particle lifting devices examined. The concentration technique gave highly variable results between treatments but still had a higher ln (x+1) of particle per hour mean than the glue lift technique (Table 5).

The tape lift surfaces were found to be stable under all treatments tested. They have a shelf life of at least six weeks and are not effected by twelve hour exposure to temperatures which might be encountered by collection devices stored in a crime scene vehicle.

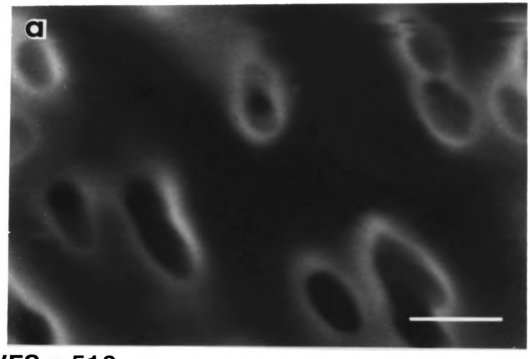
The concentration technique on the other hand, gave highly variable results between treatments. Some possible explanations which may contribute to the highly variable results (Table 7) obtained using the concentration method are as follows: 1) The concentration technique is actually a combination of two techniques; collection and concentration. Whether a decreased number of particles found per hour was due to the Vistanex surface being less efficient in collecting the particles or due to loss of particles during the concentration procedure can not be determined from this experiment.

There are at least three possible areas where GSR may be lost in the concentration method: 1) particles lost from nonadhesive 0.45 micrometer Nucleopore filter when it

was teased away from subfilter. 2) actual particles not counted due to Ba, Ca and Si contamination of subfilter. 3) particles trapped in debris and either aspirated out of concentrator or pelleted onto a filter surface.

The data, in fact, suggest that a problem may exist in the concentration procedure. If one examines the particles/hr. means of three rounds fired vs one round fired in the concentration method, the means are 0.60 and 6.1, respectively (Table 7). This was exactly opposite to the results one might expect. Going back to the actual concentration procedure employed, it was noted at the time that in six out of the seven concentrators the 0.45 micrometer Nucleopore filters could not be peeled away from the underlying Nucleopore D-79 subfilter. The filters were mounted together on an aluminum stub, carbon coated and viewed. Analysis in the electron microscope gave high background levels of Ba, Ca, Si, and K for the 0.45 um Nucleopore filters adhered to subfilters (Figure 6). The surface itself tended to pucker and charge to a degree that a reliable backscatter image was unattainable. At this point, the 0.45 um filter was dissected away from the subfilter with a razor blade and remounted on an aluminum stub. This required manipulation of the 0.45 um filter, which has no adhesive nature of its own, and it is quite likely that GSR particles were lost during this manipulation.

Figure 6. Identification of contaminating elements on a filter surface used in the concentration technique. 6a) Secondary electron image of a 0.45 um Nucleopore filter adhered to a D-79 subfilter after carbon coating. Magnification - 16,000X, bar = 1 micrometer. 6b) Energy dispersive X-ray spectrum of the filter sandwich in 6a. Vertical full scale = 512 counts, X axis from 0.0 - 15.0 KeV.



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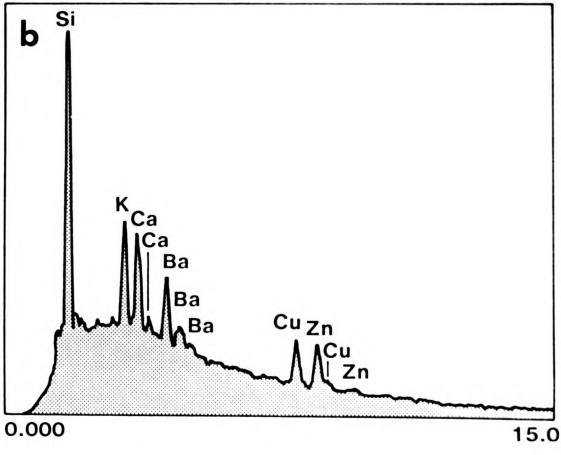

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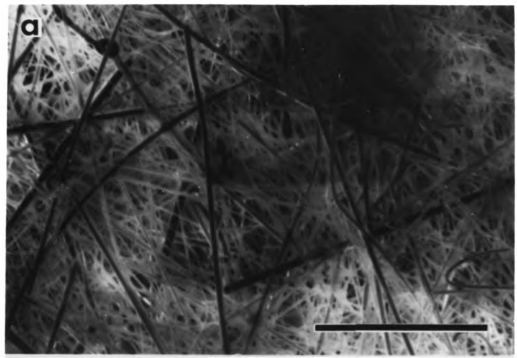
In contrast, with the concentrators used in the one round fired experiment, five out of seven Nucleopore filters were easily removable from the subfilter surface with the remaining two picking up only slight subfilter contamination.

After barium and calcium contamination was observed, a subfilter was mounted on an aluminum stub and carbon coated to determine what elements were present in the subfilter. Results of this analysis showed the presence of silicon, potassium, zinc, calcium and barium (Figure 7). This contamination compounds the problem of GSR analysis. One of the forms of GSR recognized in the Aerospace Corp. study (Wolten et al. 1977), were spheroid particles containing the elements: lead or sulfur, silicon, calcium and barium. On a 0.45 um Nucleopore filter adhering to a subfilter, such a particle would be difficult to distinguish from a pure lead or sulfur particle. Therefore, all GSR reported in the concentration method consisted of particles composed of lead, barium and antimony, unless the filter surface showed no background element contamination from adhering subfilter material.

Other investigators, using a different type of concentrator, noticed lead and barium contamination of the fifty micrometer porous polythene filter in their concentration device. Washing the filter with 20% hydrochloric acid was found to remove the contamination in

Figure 7. Identification of contaminating elements in the subfilter used in the concentration technique. 7a) Secondary electron image of carbon coated D-79 subfilter. Magnification - 300X, bar = 100 micrometers. 7b) Energy dispersive X-ray spectrum of subfilter surface in 7a. Vertical full scale = 4096 counts, X axis from 0.0 - 15.0 KeV.



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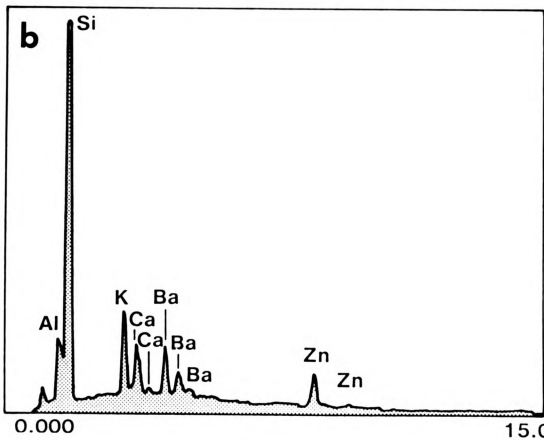

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their case (Wallace and Keeley 1979).

A third factor, which could influence the number of particles found after concentration, is the amount of debris deposited on the 0.45 um Nucleopore filter. One of the main reasons originally proposed for the concentration method was to reduce the amount of epidermal cells and other debris picked up by the collection device. Such debris may cover GSR particles present making them undetectable in the electron microscope (Ward 1982). Mr. Dennis Ward at the FBI crime lab has suggested that centrifugal force may be a critical factor depending on the amount and type of debris present on the Vistanex surface. Too low of a g-force results in material floating on the bromoform surface, which may have trapped GSR in it. Too high of a g-force may pellet debris onto the filter obscuring GSR particles (personal communication).

A varying amount of debris was found on the filter surface in the concentration technique (Fig. 8). While the present study was being conducted, an experiment comparing tape lifts to the concentration technique was performed by Zeichner et al. (1989). These researchers concluded that the build up of debris on the filter was such a problem, that direct observation of a tape or glue lift surface was preferable to concentration.

The concentration technique used in the current study was a modification of Ward's technique (Ward 1982),

Figure 8. Varying amounts of debris found on filter surface after the concentration technique. 8a) Secondary electron image of the surface of a 0.45 um Nucleopore filter after GSR concentration. A small amount of debris can be observed scattered across the center of the filter. 8b) Secondary electron image of the surface of a 0.45 um Nucleopore filter after GSR concentration. Many pieces of debris can be seen across the center of the filter. 8c) Secondary electron image of the surface of a 0.45 um Nucleopore filter after GSR concentration. The filter surface is almost totally obstructed by debris. All micrographs are at a magnification of 10X, bar = 1000 micrometers.

a
b
c

developed by Loren Sugarman at the Forensic Science Laboratory of the Orange County Sheriff-Coroner Department in Santa Ana, CA. (Appendix A). Mr. Sugarman has been able to circumvent some of the problems in the present study (personal communication). He has not noticed any contamination of the D-79 subfilters obtained from Nucleopore, which suggests the contamination of the subfilters in the current study may be a batch defect. To reduce adhesion between the 0.45 um filter and subfilter, Mr. Sugarman recommended placing the subfilter with the cross hatched surface facing up and removing the 0.45 um Nucleopore filter immediately after centrifugation. He also advised washing the filter through methanol thoroughly after the bromoform step to remove any traces of bromine on the filter surface which would give interfering backscatter signals.

The glue lift technique was found to have an inefficient particle lifting surface (Table 5). These findings were not in agreement with the observations published by the developers of the glue lift technique (Basu and Ferriss 1980).

The glue lift technique was developed by Dr. Samarendra Basu and Dr. Stark Ferriss (Basu and Ferriss 1980). It was designed to be less sticky than the tape lift surface. The reasoning was that the decreased stickiness of the glue lift surface would not collect so much interfering epidermal

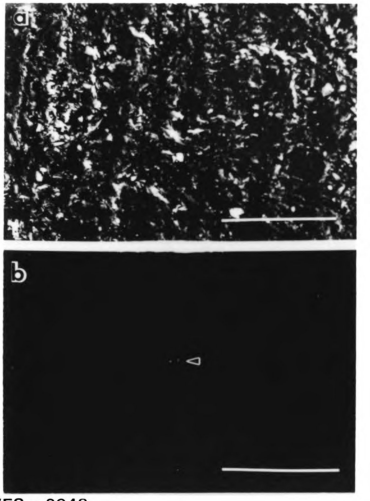
cells and other debris.

In their original paper on the development of the glue lift technique, several advantages of the glue lift surface were demonstrated compared to the tape lift surface (Basu and Ferriss 1980). Advantages included: 10 or more particles found per area searched (1.5mm diam. circle) 2) no electron beam damage to glue lift surface vs melting of the tape lift surface 3) smoother surface of carbon planchet 4) no carbon coating required.

At this point it is worth examining some differences between the two studies.

The carbon planchets used in the study by the developers of the glue lift were obtained from Ernest F. Fullam Inc. (Schenectady, NY 12301.) They were described as the "clean, polished carbon planchets (disc thickness 1/8", diameter 1/2"). In the present study carbon planchets were obtained from Ladd Research Industries Inc., specially smoothed surface carbon specimen mounts (disc thickness 5mm, diameter 15mm). At high magnification, the surface of the carbon planchets obtained from Ladd Research Industries appeared somewhat irregular (Fig. 9). Dr. Basu suggests that lack of a smooth regular surface may impair particle lifting ability (personal communication). In addition, the carbon planchets obtained from Ladd Research Industries were contaminated with tungsten particles generally of about 0.5-2.0 microns in diameter (Fig. 9). Spraying the planchets

Figure 9. Identification of contaminating tungsten particles on a carbon planchet. 9a) Secondary electron image of the surface of a carbon planchet obtained from Ladd Research Industries Inc. Magnification - 300X, bar = 100 micrometers. 9b) Backscatter electron image of the same field as in 9b. Arrowheads point to contaminating tungsten particles. Magnification - 300X, bar = 100 micrometers. 9c) Energy dispersive X-ray spectrum of a single tungsten particle. Vertical full scale 2048 counts, X axis from 0.0 - 15.0 KeV.



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with compressed air prior to applying the glue surface proved insufficient to remove all of the tungsten particles. The source of the tungsten was presumably material left behind during the cutting process at the factory. An average of nine contaminating tungsten particles per glue lift were encountered. These particles mimic GSR in the backscatter mode and lengthen analysis time.

The authors of the glue lift technique examined a minimum of four 1.5mm circles on the glue lift surface, finding an average of 58 particles per circle (Basu and Ferriss 1980).

Typically, one hour of search time, in the present study, at 300X resulted in searching approximately 15% of the total surface area of the 15mm diameter carbon planchet. This was equivalent to examining 2.85 of the 1.5mm circles described by the developers of the glue lift (Basu and Ferriss 1980).

Ammunition used in the two studies was also different. The ammunition used in the present study was Federal .38 special cal., jacketed soft point lead, 125 grain for law enforcement use. Ammunition from the same lot number (12 A) was used throughout the study. This ammunition was chosen because it gave consistently few gunshot residue particles. It was felt that this resembled actual case work conditions in a more realistic fashion than an ammunition which produces hundreds to thousands of particles. The developers of the glue lift used either "standard Winchester or

Remington ammunition" (Basu and Ferriss 1980) for the pistol loads. In the present study, Remington .38 cal., 158 grain lead ammunition was test fired from the same revolver used in this work and found to produce hundreds of particles, mainly lead in composition. The glue lift developers findings of "30-116 residues per 1.5mm diameter circle" (Basu and Ferriss 1980), on the glue lift surface was consistent with the Remington ammunition tested. However, the finding that "a typical 1/2" diameter tape-lift disc may contain from 2-10 observable GSR." (Basu and Ferriss 1980), with one round fired was not consistent with the Remington ammunition tested. In support of the finding of few GSR on a tape lift surface, the authors quote a table in the work by Sild and Pausak (1979), where it was mentioned that "firing two shots with a .38 cal revolver, they recovered slightly more than 8 GSR and 20 lead particles from a one inch diameter "tape-lift" disc (Basu and Ferriss 1980). If one examines Table I. in Sild and Pausak (1979), one can also find a test firing of 2 shots with a .38 cal. revolver where a single sweep (magnification 1000X) shows more than 20 particles of lead and greater than 20 particles of GSR (Pb+Sb+Ba) are found on the whole stub.

In the original study of the glue lift technique, Basu and Ferriss cite the beam damage that occurs on the tape lift surface. They demonstrated this with a micrograph (Basu and Ferriss 1980 Fig. 2-f) depicting "a lead particle

disappearing into a cavity on transfer tape, created by the bombarding electrons." . The authors went on to discuss particles imbedding themselves and disappearing into the melted tape surface.

In the present study, where hundreds of particles of various composition were observed and spectra obtained, no particle was ever seen to "disappear into the melted surface of the tape". Electron beam damage to the tape surface did occur and usually appeared as a crater with surrounding folds around the particle (Figures 10a & 11a). The only time the tape lift surface was seen to crack or melt severely was if it was not coated with enough carbon initially (Fig. 12). This problem was easily remedied by applying another carbon coat. The thickness of a single carbon coat was typically in the range of 35-40nm. (Appendix B).

Another way to induce electron beam damage is by using excessive beam current. In the work by Basu and Ferriss (1980), the beam or specimen current used was not mentioned. The emission current was listed as 100 microamps but this gave no information as to the current which the specimen is encountering. In the present study, a beam current of 550 picoamps was used. This was measured using a Faraday cup inserted after the final aperture. The tape lift surface was found to be stable under these conditions.

Older energy dispersive x-ray analysis systems may

Figure 10. Electron micrographs of small GSR particles on various surfaces. 10a) Secondary electron image of GSR on a tape lift surface. 10b) Backscatter electron image of GSR particle in 10a. 10c) Enlargement of GSR particle in 10a. 10d) Secondary electron image of GSR on a 0.45 um Nucleopore filter surface. 10e) Backscatter electron image of GSR particle in 10d. 10f) Enlargement of GSR particle in 10f. 10g) Secondary electron image of GSR on a glue lift surface. 10h) Backscatter electron image of GSR particle in 10g. 10i) Enlargement of GSR particle in 10g. Magnification for micrographs a,b,d,e,g and h - 300X, bar = 100 micrometers. Magnification c,f and i - 12,000X, bar = 1 micrometer.

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Figure 11. Electron micrographs of large GSR particles on various surfaces. 11a) Secondary electron image of GSR on a tape lift surface. 11b) Backscatter electron image of GSR particle in 11a. 11c) Enlargement of GSR particle in 11a. 11d) Secondary electron image of GSR on a 0.45 um Nucleopore filter surface. 11e) Backscatter electron image of GSR particle in 11d. 11f) Enlargement of GSR particle in 11f. 11g) Secondary electron image of GSR on a glue lift surface. 11h) Backscatter electron image of GSR particle in 11g. 11i) Enlargement of GSR particle in 11g. Magnification for micrographs a,b,d,e,g and h - 300X, bar = 100 micrometers. Magnification in c - 2,400X, bar = 10 micrometers. Magnification in f - 2,700X, bar = 10 micrometers.
Magnification in i - 1,200X, bar = 10 micrometers.

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Figure 12. Effect of insufficient carbon coating on a tape lift surface. 12a) Secondary electron image of tape lift surface cracking and charging due to beam damage from insufficient carbon coating. 12b) Secondary electron image of the same tape lift stub after a second carbon coating was applied. Magnification of both micrographs - 30X, bar = 1000 micrometers.

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b
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require higher beam currents when accumulating EDX spectra. An example of this was observed during the present study at the Center for Electron Optics. The Tracor Northern TN2000 EDX system required a beam current of approximately 1000 picoamps to accumulate a spectrum with a 30% dead time for a given GSR particle. Keeping the same detector but changing the hardware to a newer TN5500 EDX system resulted in the accumulation of an EDX spectrum at 500 picoamps with a 30% dead time on the same GSR particle.

Perhaps the developers of the glue lift technique were using an older instrument requiring a high beam current or had insufficient carbon coating on their tape lift samples. This would explain the observed melting of the tape surface and the overall diminished particle counts, as particles "disappeared" from view into the melted surface.

The procedure for sampling the hand with the glue lift disc was different than the hand sampling procedure using the tape lift disc. The developers of the glue lift maintain that the hand should be touched only 5 times along the thumb, web and forefinger for sampling the "back" of the shooters hand (Basu and Ferriss 1980). Whereas, authors using the tape lift method recommend touching the entire area of thumb, web and forefinger (about 12 touches) or until the stickiness of the tape is lost (Matricardi and Kilty 1977, Nesbitt et al. 1976, Wolten et al. 1977, Wolten et al. 1979b). In the present study, perhaps the tape lift

picked up more particles because a greater surface area of the hand was sampled.

This possibility was examined by collecting GSR from the hand using a glue lift surface first and dabbing the hand twelve times along the thumb, web and forefinger. This collection was then followed by a tape lift collection along the same area. The data in Table 9 indicated that when the surface area sampled was held constant the glue lift remained a less efficient particle lifting device compared to the tape lift.

SUMMARY AND CONCLUSIONS

OBSERVED ADVANTAGES AND DISADVANTAGES OF EACH COLLECTION METHOD

Besides examining collector particle lifting efficiency for a variety of conditions such as number of rounds fired, temperature and storage time, a table of observed advantages and disadvantages for each collection method was developed (Table 10).

The tape lift method for GSR collection has the primary advantage of having an efficient particle lifting surface, as previously discussed. The tape itself is inexpensive and the collection devices are simple to construct. The adhesive surface was found to be stable, i.e. particle lifting ability was not decreased significantly, under all conditions tested. The tape lift surface gave a good secondary electron image, which is important for photographing particles (Figs 4a, 4c & 5a, 5c), especially when using an instrument that has a backscatter detector that does not operate at the normal TV scanning rate (not recommended).

Disadvantages of the tape lift method are few. The surface requires a carbon coat. Depending on the size stub used, there is a relatively large surface area to be scanned. Debris collected from the hand may hide GSR

Table 10. Observed advantages and disadvantages of GSR collection methods tested.

Method	Advantages	Disadvantages
Tape Lift	Efficient particle lifting surface	Requires carbon coat
	Inexpensive	Large surface area
	Simple to prepare	Skin debris may hide particles
	Temperature stable	
	Stable shelf life at least 6 weeks	
	Good secondary image	
Glue Lift	Requires no carbon coat	Inefficient particle lifting surface
	Easy to prepare	Contaminated with tungsten particles
	Picks up less debris	Carbon planchets expensive
	Fair secondary image	Large surface area
		Skin debris may hide particles
Concentration	Separate debris from GSR	Requires carbon coat
	Small surface area	Collection efficiency variable
	Pre-made collectors and concentrators can be purchased	expensive
		2hr. Processing time
		Subfilter contamination
		Filters stick together
		Poor secondary image

particles beneath it.

The chief advantage of the glue lift is that the carbon coating step may be skipped. The devices are also quite simple to construct. The secondary image was not optimal in the present case due to the roughness of the carbon planchets obtained from Ladd Research Industries (Figs 10g, 10i & 11g, 11i). Their theoretical advantage is that the surface is less sticky and therefore picks up less debris from the hand.

Unfortunately, the glue lift surface did not pick up much GSR either. It was found to be an inefficient particle lifting surface. The stability of the glue lift surface to temperature and storage could not be determined due to the minimal number of GSR found on the glue lifts for all treatments tested. The carbon planchets themselves were moderately expensive and were found to be contaminated with interfering tungsten particles. The problem of picking up debris from the hand was reduced with the glue lift surface but not entirely eliminated. The surface area to be searched is the same as in the tape lift which is relatively large. Typically, one hour of search time at 300X resulted in searching approximately 15% of the total surface area of the 15mm diameter carbon planchet.

The concentration technique has the potential of separating GSR from debris. Pre-made collection devices may be purchased from Kinderprint Co. Inc. or made by the

investigator at minimal cost. The total surface area to be searched is reduced to the point where a manual search of the entire filter is possible in less than one hour, provided that there are not a lot of interfering particles of high atomic number.

In the present study, the concentration method was found to give highly variable results. Particle lifting efficiency and stability of collection surface could not be determined due to several factors contributing to particle loss. As discussed previously and as listed in Table 10. those factors were: 1) contamination of subfilter with barium, calcium and silicon 2) particle loss due to manipulation of filter surface 3) particle loss due to aspiration of particles trapped in debris or particles trapped in debris on filter surface.

In addition to above factors, the concentrators themselves were relatively expensive items (Appendix A). In order to achieve a decent secondary electron image, the 0.45 um Nucleopore filter had to be carbon coated rather heavily. Even under these conditions, discriminating GSR particles from the background in order to obtain a photograph was difficult at best (Figs. 10d, 10f & 11d, 11f). An extensive methanol wash must also be used to remove bromoform from the filter to reduce interference in the backscatter mode. Finally, one must consider the additional time required to concentrate the samples. This was approximately two hours

to prepare six samples.

THEORETICAL OPTIMAL COLLECTION DEVICE

All of the methods examined had their advantages and disadvantages (Table 10). Perhaps the optimal collection device would be one that combines advantages of all three collection techniques. One would like to have a surface that does not require carbon coating, as in the glue lift technique. That surface would ideally be polished smooth and flat, with no contaminating elements of high atomic number. The surface should be coated with a substance that has the stickiness of 3M 465 adhesive transfer tape. If the collector was viewed directly in the electron microscope and found to have too much debris on its surface, then one would like to be able to take that same surface and apply the concentration technique to it. The subfilter of the concentrator should be free of contaminating elements of high atomic number. The final filter surface should give a better secondary image than is currently obtained on 0.45 um Nucleopore filters. The collection device should be relatively inexpensive and stable to conditions of temperature and time (i.e. long shelf life).

In actuality, the proposed collection device could use a polished graphite circular 15mm wafer as the sample surface. This wafer could be coated with diluted Vistanex adhesive which is stickier than rubber cement. If, after

viewing the sample initially, concentration was deemed appropriate a concentration device with a larger bore diameter to accommodate the 15mm wafer could be used. The entire concentration device would then be sonicated to remove and solubilize the Vistanex surface at which point the wafer could be removed and concentration could proceed as described in Appendix A. A final filter of the same diameter but of a wider pore spacing which is more conducive to carbon coating could be used as suggested by Wallace and Keeley (1979).

At the moment, cost is the initial stumbling block. Highly polished graphite planchets are available but are extraordinarily expensive. It would be interesting to obtain some of these highly polished planchets and see to what degree particle lifting efficiency could be improved over the current collection devices.

CHAPTER THREE

THE "GUN CONTROL" EXPERIMENT

INTRODUCTION

The purpose of the hand and gun control collections was to determine whether there was a certain amount of "background" GSR present after washing of the hand or working the action of a freshly fired, but cleaned gun. The expectation of the researcher was that these collection devices would show low levels of GSR. After the randomly numbered devices collected as part of the experiments in Chapter Two of this thesis had been analyzed in the electron microscope, the numbers were decoded and the hand and gun control data were compiled. The hand controls gave a characteristically low GSR particle/hr. mean (Table 6).

The gun controls however, resulted in a consistently high number of GSR particles being found on the hand (Table 6). This result was rather unexpected, as finding GSR on the hand after pulling the trigger on a cleaned unloaded weapon had never been previously reported in the literature.

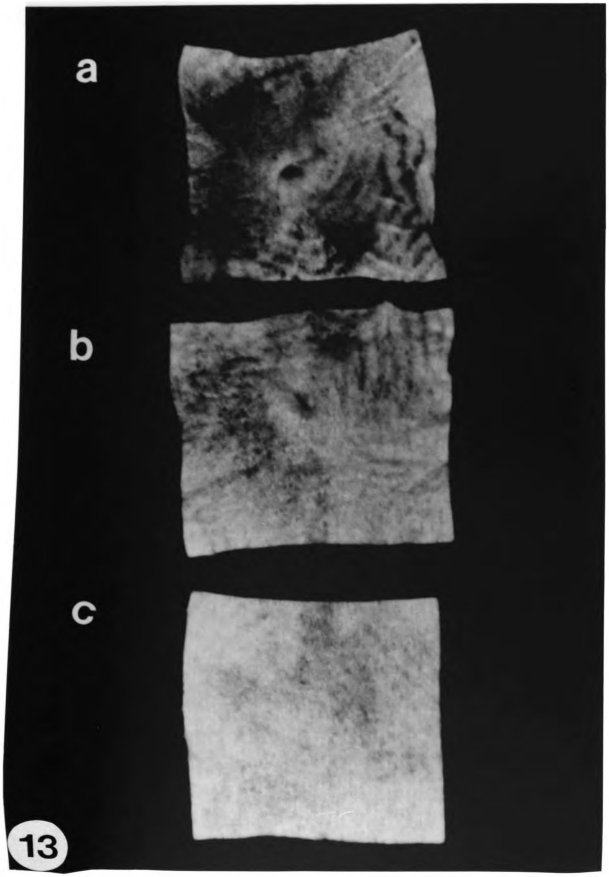
Since the gun control finding was unexpected and rather startling, a more rigorous experiment was designed in an attempt to reproduce the gun control observations. In the original gun control collections, the revolver was cleaned in the same room where the test firing had taken place. This room was equipped with an exhaust fan that was kept running while shooting and cleaning took place. It is

possible that GSR was settling out of the air onto the revolver during the cleaning process. It has been demonstrated that a collection device left exposed in a firing range behind the shooter will collect GSR (White and Owens 1987). In addition, in the original gun control observations, the person doing the repetitive firing was also the person who dry fired the weapon for the gun control collection. In the redesigned gun control experiment these potential factors were controlled.

MATERIALS AND METHODS

In the redesigned gun control experiment, one investigator fired three rounds of Federal ammunition (.38 special cal. copper jacketed 158 grain bullet). The revolver (S&W Model 10-8) was immediately removed from the firing room and taken to a separate room where it was cleaned, as described previously. Swabs from the cleaning operation were kept and photographed to demonstrate the diminished powder residue by the third swabbing (Fig. 13). A second investigator, not having fired the weapon or been in the firing room, washed his hands and dry fired the revolver three times. His hand was then sampled as described previously with collection devices of the 3M 465 adhesive transfer tape type. This procedure was repeated five times along with one hand control sample taken from the second investigator's freshly cleaned hands. Collection devices were then assigned random numbers, carbon coated and analyzed in the electron microscope.

Figure 13. Gun cleaning patches from the modified gun control experiment. 13a) Solvent soaked patch used in the initial cleaning of the barrel, chambers and interior of the revolver after three rounds had been fired. 13b) Second solvent soaked patch used to clean barrel, chambers, interior and exterior of the revolver. 13c) Final dry patch used to clean barrel, chambers, interior and exterior of the revolver.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RESULTS AND DISCUSSION

As in the original experiment a high amount of GSR was found on the investigator's hand after dry firing the cleaned weapon (Table 11). In fact, there was no significant difference with a 95% confidence interval, in a comparison of the mean number of particles found per hour between three live rounds fired and working the trigger mechanism on the unloaded, cleaned weapon three times (Table 11).

The finding that significant amounts of GSR remain on or in the weapon after cleaning may help explain why no significant difference was observed between three rounds and one round fired for the tape lift method (Table 6). If cleaning the weapon results in high amounts of GSR still associated with the cleaned weapon, then differences between one or three rounds fired may be less pronounced than they would be if all GSR had been removed in the cleaning process.

Other researchers described cleaning the firearm between firings (Basu et al. 1984, Brihaye et al. 1982, Gansau and Becker 1982, Wolten et al. 1979a), but made no mention of collecting a sample from the hand off a cleaned "dry fired" weapon.

There was one case where the possibility of finding GSR on a cleaned weapon was mentioned (Basu et al. 1984). This

Table 11. Comparison of GSR found on the hand between three live rounds fired and "dry firing" an unloaded, cleaned weapon three times.

	Number of GSR particles found on the hand		
Treatment	Lower 95% C.L.	Mean	Upper 95% C.L.
3 rounds fired	10.8	7.9	5.7
"dry fired" 3 times	14.3	8.4	4.8

appeared in a study of suicide reconstructions where the authors attempted to determine whether a suicide victim used a cleaned weapon or an unclean weapon in the shooting. The authors proposed that gun cleaning oils and grease may retain residues and, therefore, "dry cleaned" the gun with patches soaked in methanol to remove the contaminating particles of GSR before test firing for reconstruction purposes. The authors then concluded: "one is able to estimate by a comparison of the GSR densities if the suicide victim's hands have had excess residues over the amounts obtained from the test shooter's hands. The presence of these excess residues on the victim's hands was the indication that he used an unclean gun." (Basu et al. 1984).

The observation that gun cleaning oils and grease retain GSR particles was significant and pertinent to the gun control findings in the present work. The conclusion that excess residue on the suicide victim's hands compared to the test shooter's hands was indicative of the victim having used an unclean gun was questionable however, since lay-people do not normally clean their guns with methanol soaked patches to remove gun cleaning oils and grease that may retain GSR.

SUMMARY AND CONCLUSIONS

The finding of large amounts of GSR on the hand from dry firing a cleaned weapon has serious implications in the interpretation of finding GSR on a suspect's hand. Use of the SEM equipped with EDX allows the investigator to conclude that certain particles removed from the hand are gunshot residue and nothing else, but the question still remains as to how the GSR ended up on the hand.

The answer to the question: "How did it get there?" has always been the Achilles heel of GSR analysis. One would like to say that the only way for GSR to appear on a suspect's hand is if the suspect had fired a gun. Unfortunately, as the present study demonstrates, that may not always be the case. The results of the present study indicate that one must proceed with extreme caution in making inferences from a positive GSR finding on the hand.

Future experiments should examine the following questions: 1) How well does the cleaning of the weapon with methanol soaked patches as described by Basu et al. (1984) work? 2) If GSR can be removed from a cleaned weapon using the above procedure can significant differences between one round and three rounds fired be determined for the tape lift method? 3) Is GSR picked up by simply handling a cleaned weapon without operating the trigger mechanism?

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APPENDIX A

The following material in Appendix A was distributed to Crime Labs throughout the country by Kinderprint Co., Inc. P.O. Box 16, Martinez, CA. in the Spring of 1988.

APPENDIX A

THE CONCENTRATION AND ISOLATION OF GUNSHOT RESIDUES FOR PARTICLE ANALYSIS

BY

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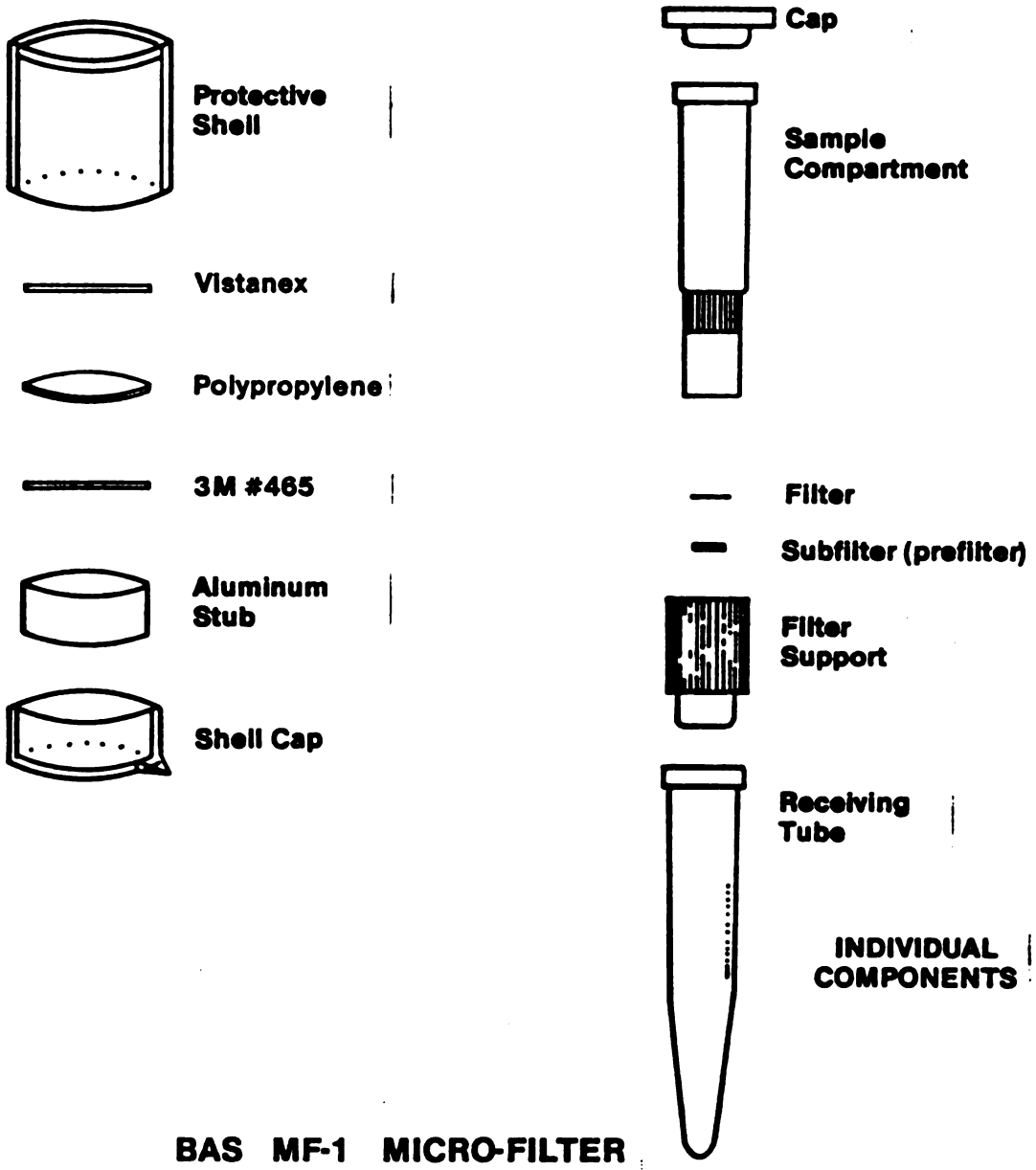
ORANGE COUNTY SHERIFF-CORONER DEPARTMENT
SANTA ANA, CALIFORNIA

An efficient, cost effective method of concentrating GSR particles for SEM/EDX analysis has been developed, using commercially available, disposable materials.

A 25mm diameter adhesive sample is concentrated to a 5mm area on a polyester membrane filter having a 0.4 micron pore size. Particles are released from an adhesive coated disc of polypropylene by dissolving the adhesive with trichloroethylene in a microcentrifuge filter tube. Following centrifugation, the light-particle fraction is aspirated from the top of the solvent. The density separation is repeated using bromoform. High density particles are deposited onto the filter by centrifuging the unaspirated solvent through the membrane. Sample preparation requires 15-20 minutes.

Recovery efficiency, evaluated by FAAS, was determined relative to the total Pb and Sb from the aspirate and filtrate fractions.

The concentration procedure reduces the searching area to 4% of the original surface area and the density separation removes the organics and low density debris, minimizing the burying of GSR under other particles. Approximately 90% of the total GSR is recovered on the membrane. The remainder of the GSR is found in the aspirated fraction.



BAS MF-1 MICRO-FILTER

SAMPLE PREPARATION:

1a) Remove the polypropylene disc (with Vistanex) from the collection stub.

b) The disc should be curled, adhesive inward, and inserted into the sample compartment of the filtration tube.

c) The cork should be firmly seated in the base of the filter section.

2) Add 2 ml Hexane to nearly fill tube. Allow 5 minutes for the adhesive to dissolve. Cap the filter tube and shake vigorously to assist in dissolution.

3a) Remove the cap and place the filter tube into a disposable test tube so that the system is resting on the cork.

b) Centrifuge at moderate rate for 5 minutes to speed the separation. The centrifuge used for this work has a radius of 12 cm to the bottom of the tube and is spun at 1,100 rpm.

4) Aspirate off the top 1.5 ml of solvent (approximately 80% of the depth due to conical shape of the tube) to remove the floating debris.

5a) Refill the tube with bromoform (density 2.89) approximately 1.5 ml.

b) Recap and shake vigorously to mix bromoform and Hexane.

c) Return the filter to test tube and centrifuge for 10 minutes at 1,100 rpm.

6a) Aspirate off the top 1.0 ml of solvent mixture with the floating debris.

b) Pull the cork, replace the receiving tube on the filter device and centrifuge the remaining solvent through the filter until dry (approximately 5 minutes at 1,100 rpm).

7) Two milliliters of OmniSolve[1] grade MeOH are added to the sample compartment and centrifuged through the filter. (approximately 5 minutes at 1,100 rpm).

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[1]Filtered for particulates (0.2 microns).

8) The membrane is removed with clean forceps, mounted onto an SEM stub and carbon coated for examination.

The entire preparation procedure requires about 40-60 minutes. Samples are generally prepared in groups of four, so preparation time averages between 10-15 minutes per sample.

Material costs run approximately $3.00 per sample excluding chemical costs.

Material Requirements for Gunshot Residue Concentration

Aluminum Mounts: (.48 each)

Ted Pella Co.
Box 510
Tustin, CA 92681
(714) 863-0666

Offices and Warehouse:
16812 Millikin Ave.
Irvine, CA 92714
(714) 557-9434

Cat. #16279-Specimen Mounts, 15/16" X 3/8", aluminum. $480.00/1000

Covers and Caps: (.102 each)

Order:
Riekes Container Co.
6270 Caballero Blvd.
Buena Park, CA 90620
(714) 522-8740

Supplier:
Brockway
Flex Products
445 Industrial Rd.
Carlstadt, NJ 07072
(201) 933-3030

1" X 1-1/2" ol Shell Containers and 1" PE Plugs, natural color. $102.00/1000

Centrifuge Tube: (2.085 each)

Bioanalytical Systems Inc.
2701 Kent Ave.
Purdue Research Park
W. Lafayette, IN 47906 (317) 463-4527

MF-1 Microfilter. $29.00/pk. of 12

Membrane Filters: (.64 each)
Subfilter Supports: (.13 each)

prices vary with order size

Nucleopore Corp.
7035 Commerce Circle
Pleasanton, CA 94566 (415) 463-2530

Non-stock item: Polyester Membrane, 8.3mm dia., 0.4um pore. $64.00/100
Non-stock item: Subfilter, 8.3mm dia., D79 type. $13.00/100 in bulk

Culture Tubes: (.056 each)

16X 100mm disposable. Typically priced at $14.00/250

Corks: (.052 each)
XXXX Quality or better. $26.00/500
5.5mm-8.0mm size 00 from Thomas Scientific (6-8mm std. size is too big) 4mm-6mm size 000 may be sufficient.

Teflon Sealing tape: Insignificant price)

Available at hardware stores.

Centrifuge:
Clay Adams Dynac II with timer and tachometer.
Horizontal head with 6" radius to bottom of tube.

Total Price: $3.54 per sample excluding chemical costs.

Chemicals: **Prices vary with supplier and size of order**
Hexane
Purified Bromoform
Methanol. OmniSolv grade from EM Science. Prefiltered for particulates.

APPENDIX B

Calculation of thickness of Carbon Coating[1]

Carbon thickness based upon the premise that a given mass of carbon evaporates uniformly to cover a spherical target (e.g. the bell jar). The basic equation for this assumption is:

$$W = \frac{M}{4\pi R^2}$$

Where: W = weight in g/cm^2 on the surface

M = mass of the evaporant

R = radius or evaporant/specimen distance

W may also be expressed = t (thickness) x p (density in g/cm^3)

t may be considered = $\frac{W}{P}$

Typical carbon coat:

$$t = \frac{W}{P} = \frac{\frac{M}{4\pi R^2}}{P}$$

M = 4 mg

P = 9 g/cm^3

R = 10 cm

$$\frac{\frac{4.0 \times 10^{-3}\ g}{(4\pi)(10\ cm)^2}}{9\ g/cm^3} = \frac{3.183 \times 10^{-6}\ g/cm^2}{9\ g/cm^3}$$

$$= 3.5 \times 10^{-7}\ cm$$

$$= 35\ nm$$

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[1]Taken from: Exercises in Electron Microscopy, a laboratory manual for biological and medical sciences. Hooper, G R., Baker, K. K., and Flegler, S. L. (1979). Center for Electron Optics, Michigan State University, E. Lansing, MI 48824.

APPENDIX C

1988 GUNSHOT RESIDUE ANALYSIS SURVEY

1. Please circle the type/types of analysis currently being used in your laboratory on gunshot residue (GSR) samples.

A) NAA B) FAAS C) SEM-EDX D) Other (Please describe)

_____

_____

2. Approximately how much time per week is devoted to GSR analysis _____?

3. Approximately how much time is required per analysis

_____

_____

4. How frequently is your current method for analysis of GSR challenged in court and on what grounds?

_____

_____

_____

_____

_____

The following questions need only be answered by laboratories equipped with an SEM.

5. Please indicate the make and model of your SEM.

_____

6. If your microscope is equipped with X-ray analysis capability, please indicate make and model.

_____

7. If possible, please indicate how old the above instrument is. _____

8. In the past year please estimate the number of weeks the SEM-EDX was "down" due to mechanical or electrical failure

_____

_____

9. If examining GSR by SEM-EDX, please indicate the sample collection and processing technique used most frequently.

_____ A) The Vistanex adhesive lift, followed by sample concentration.

_____ B) A tape lift method.

_____ C) A glue lift method.

_____ D) Other (Please explain)

_____

_____

_____

10. Please indicate the minimum number of particles analyzed to confirm gunshot residue in your lab.

_____

11. For GSR samples being analyzed by SEM-EDX:

A) Approximately how much time per week is devoted to this type of analysis _____?

B) Approximately how much time is required for a positive (evidence of GSR) analysis _____?

12. Please describe any recurring problems and/or solutions you have found in either collection or analysis of GSR by the SEM-EDX technique.

_____

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