

Current Methods in

FORENSIC
GUNSHOT
RESIDUE
ANALYSIS

Current Methods in

**FORENSIC
GUNSHOT
RESIDUE
ANALYSIS**

A. J. Schwoeble • David L. Exline



CRC Press

Boca Raton London New York Washington, D.C.

Library of Congress Cataloging-in-Publication Data

Schwoeble, A. J.

Current methods in forensic gunshot residue analysis / by A.J. Schwoeble,
David L. Exline.

p. cm.

Includes bibliographical references.

ISBN 0-8493-0029-0

1. Forensic ballistics. I. Exline, David L. II. Title.

HV8077.S34 2000

363.25'62—dc21

00-030353

CIP

This book contains information obtained from authentic and highly regarded sources. Reprinted material is quoted with permission, and sources are indicated. A wide variety of references are listed. Reasonable efforts have been made to publish reliable data and information, but the author and the publisher cannot assume responsibility for the validity of all materials or for the consequences of their use.

Neither this book nor any part may be reproduced or transmitted in any form or by any means, electronic or mechanical, including photocopying, microfilming, and recording, or by any information storage or retrieval system, without prior permission in writing from the publisher.

The consent of CRC Press LLC does not extend to copying for general distribution, for promotion, for creating new works, or for resale. Specific permission must be obtained in writing from CRC Press LLC for such copying.

Direct all inquiries to CRC Press LLC, 2000 N.W. Corporate Blvd., Boca Raton, Florida 33431.

Trademark Notice: Product or corporate names may be trademarks or registered trademarks, and are used only for identification and explanation, without intent to infringe.

© 2000 by CRC Press LLC

No claim to original U.S. Government works

International Standard Book Number 0-8493-0029-0

Library of Congress Card Number 00-030353

Printed in the United States of America 1 2 3 4 5 6 7 8 9 0

Printed on acid-free paper

Preface

Gunshot residue (GSR) examination is a specialized discipline of forensic science. Techniques for examining GSR have evolved from simple color tests to modern analytical methods. This book details current methods of GSR analysis for professionals who utilize this type of evidence in their work and introduces the subject to laypeople interested in learning about this ever-changing field.

The concept of this text was formulated with current methods of GSR in mind. A comprehensive text on the examination of GSR was compiled more than 20 years ago in a publication called the Aerospace Report No. ATR-77(7915)-3. To date, the Aerospace Report is the most comprehensive text available about GSR. Since that time, advancements have been made in the way GSR evidence is examined in laboratories worldwide, and many laboratories have modified the classification of certain particles derived from this type of evidence. This book is not meant to replace the extensive research compiled by the Aerospace Report, but rather to supplement that report and many other quality research papers that have been published over the past 20 years.

This text concentrates on certain areas that we consider most important to examiners in the law enforcement community and students of criminal justice and forensics. Chapter 1 discusses the significance of GSR evidence, collection criteria, and general topics. Chapter 2 examines particle formation and presents a current classification scheme for GSR evidence. The classification of particles is an important aspect of GSR evidence that is evolving as research in this area continues. A general overview of scanning electron microscopy energy dispersive spectroscopy (SEM/EDS) is presented in some detail in Chapter 3. This chapter will educate both bench examiners and students about this powerful method of analysis, now at the forefront of GSR examination. General theory, computer-controlled scanning electron microscopy (CCSEM), and variable pressure scanning electron microscopy are discussed.

Chapter 4 details a study of “plume” concentrations. The importance of how GSR is deposited on the hands of shooters with various types of firearms is a significant aspect of GSR interpretation. The various amounts and areas

of deposition of GSR for firearms such as revolvers, pistols, rifles, and shotguns are explained and compared. A possible mechanism to estimate particle fallout is also described.

No explanation of GSR would be complete without a general overview of commonly used firearms and ammunitions. In Chapter 5 we attempt to educate the reader on the general types of firearms used today and the components of commonly used ammunition types. Chapter 6 summarizes general guidelines for note taking, report writing, and courtroom considerations for the expert witness.

This book presents the reader with an overview of current methods of examination and interpretation used by the authors and examiners in the law enforcement community. Our hope is that the reader will combine the knowledge provided in this book with past and current literature about GSR examination, and thus continue the evolution of this valuable area of forensic science.

The Authors

A. J. Schwoeble is Manager of Forensic Sciences and Special Projects, responsible for forensic analytical services and for designing, implementing, and reporting specialized testing programs for RJ Lee Group, Monroeville, PA. At RJ Lee Group, Mr. Schwoeble played a key role in developing latent fingerprint technology and the adhesive-lift technology used for sampling skin and fabric for trace evidence applications and for surface and indoor ambient air particulate sampling. He was a principal architect for computer software to perform gunshot residue (GSR) analysis. He is presently consulting with a Swiss law enforcement agency for the analysis of GSR produced from foreign manufactured ammunition, and conducting the examination of clean fire primers of U.S. manufacturers.

Mr. Schwoeble has consulted with the FBI and other law enforcement agencies on automated microscopy and sample collection techniques for the analysis of GSR and other trace evidence. He performs GSR analysis and particle analysis for over 150 law enforcement agencies and attorneys in the United States, Australia, and Switzerland. His forensic investigations incorporate the use of optical, scanning electron, and transmission electron microscopy, atomic absorption (AA), and micro-FTIR (Fourier Transform Infrared Spectroscopy). Mr. Schwoeble has qualified as an expert for GSR analysis in several states. He has been an invited speaker at meetings of the ICEM-14, ENFSI, MAFS, SAFS, CAFS, and MAAFS.

Prior to joining RJ Lee Group in 1986, Mr. Schwoeble acquired over 25 years of research and materials analysis experience at the Naval Research Laboratory, Washington, D.C., the United States Steel Research Center, Monroeville, PA, and Allegheny Ballistics Laboratory, Pinto, WV. He is the author of numerous publications. He was the 1984 recipient of the ASM Henry Marion Howe Award as the author of the published paper of highest merit in metallurgical transactions.

David L. Exline is a forensic scientist with RJ Lee Group, where he examines GSR, hairs, fibers, paints, and other forensically significant trace evidence. Before joining RJ Lee Group in 1998, Mr. Exline was a forensic scientist with the Anne Arundel County Police Department Crime Laboratory, Millersville,

Acknowledgments

We wish to thank the following individuals:

Dr. Richard Lee, for his encouragement, technical support, and generosity in the use of the laboratory and instrumentation, and for giving us a free hand to conduct research and experimentation

William Powers, Sr., for his contributions in test firings and his invaluable expertise in firearms and ammunition, which is evident throughout this text

Hank Beebe of RJ Lee Group, Inc. for the engineering expertise he brought to all aspects of our analyses using scanning electron microscopy (SEM)

Dr. Stephen Kennedy of RJ Lee Group, Inc., for his insight and contributions to the SEM chapter

Dr. Fred Schamber of RJ Lee Instruments, for his input and editing of the SEM material

Keith Wagner of RJ Lee Group, Inc. for his contribution dealing with GSR particle fallout calculations from discharged firearms

Cindy Lodor, for her invaluable behind-the-scene organizational abilities

Hank Lentz, for the immense time he spent with GSR experts around the country, whose input he then used to create the software for automated GSR analysis

Josh Newlin for his expertise in graphics and photography

Jim Nesbitt and Ray Callihan for their direction and participation in the production of the video analysis of plume concentrations described in this book

The team members of RJ Lee Group, Inc. for their efforts and support in the test firings of multiple weapons and ammunition studies

The authors would also like to express their appreciation to Dennis Ward and John Krall of the FBI, Kenneth Culbreath of the NCSBI, and Joseph Powell of the SCLED for their consultation and aid during the plume study.

To our wives, Nancy Schwoeble and Jennifer Exline

Their patience and support allowed us to spend many nights and long hours compiling data, writing, and organizing the contents of this book, which took us away from family life for extended periods of time

Table of Contents

1 Gunshot Residue as Forensic Evidence

- Introduction
- Value of GSR as Trace Evidence
- Degradation of GSR
- Transfer of GSR
- Specific Areas of GSR Deposition
- Collection of GSR Evidence
- General Crime Scene Considerations
- Examination of GSR
- References

2 Explanation of Gunshot Residue

- Particle Formation
- Classification of GSR Particles
 - Are All GSR Particles Spherical?
 - Shape and Texture of GSR
 - Morphologies of Known GSR Particles from Test Firings
- Uniqueness of Composition
- Terminology
- Environmental Considerations in GSR Examination
- References

3 Methods for Examination of Gunshot Residue

- Principal Techniques
- Scanning Electron Microscopy/Energy Dispersive Spectroscopy
 - Theory of the SEM
 - Features of the SEM
 - How the SEM Works
 - The Column
 - Column Issues

- Beam/Sample Interaction
- Detectors
 - Electron Detectors
- Imaging
- Imaging Considerations
- X-Ray Detectors
- X-Ray Artifacts
- Working Distance
- EDS Considerations
- Automated SEM Analysis
- Variable Pressure SEM
 - Case Study 1
 - Case Study 2
 - Case Study 3
 - Case Study 4
 - Discussion of Variable Pressure SEM
- Other Methods of Analysis
 - Color Tests
 - Bulk Methods of Analysis
 - Graphite Furnace Atomic Absorption Spectroscopy (GFAAS)
 - Neutron Activation Analysis (NAA)
 - Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS)
 - Capillary Electrophoresis (CE)
- References

4 A Study of Plume Concentrations

- The Plume Study
- Summary of Results
- Particle Fallout
- Conclusions
- References

5 Ammunition

- Firearms
 - Rifles
 - Shotguns
 - Pistols
 - Cartridges

- The Cartridge Case
 - Rimfield Cartridge
 - Centerfire Cartridge
 - Cartridge Casing
- Bullet
- Powder Charges
- Primer
- Head Stamps
- .22 Caliber Ammunition Composition
- Ammunition Composition Studies
- GSR Studies: U.S. Manufacture Pistol Primers
- General Observations from Test Firings
- Revolvers vs. Semi-Automatics
- References

6 Documentation of Evidence and Testimony

- Note Taking
- Report Writing
- Chain of Custody
- Expert Testimony
 - General Questions and Possible Explanations
 - Expert Qualifications
 - Background Questions

Glossary

Bibliography

Gunshot Residue as Forensic Evidence

1

Introduction

Forensic science may be defined as the application of various scientific disciplines to aid the criminal justice system. Gunshot residue (GSR) evidence is an area of forensic science that is often underestimated and underappreciated by the forensic community. It is certainly one of the most underutilized types of physical evidence. The staggering increase in firearms-related offenses in the U.S. has intensified the importance of rapid and accurate GSR analysis. In a recent report, the Federal Bureau of Investigation (FBI) cites firearms as the weapons of choice in over 30% of 1.7 million incidents of violent crime.¹

This book was written in response to the growing realization of the significance of GSR as evidence and the lack of textbooks that discuss current developments in this area of forensics. Despite advancements in analytical techniques and adjustments to the classification of GSR particles, no text that discusses these developments has been produced. In particular, this book provides a unique resource to help examiners at every level better understand current methods of analysis. The book will also aid experienced examiners by highlighting other areas that have been refined since guidelines were set forth by the Aerospace Report of 1977.² The Aerospace Report was a landmark study of GSR, and the information it provided to the forensic community was invaluable to GSR examiners. Since that time significant developments have occurred in the area of GSR analysis. For example, methods such as computer-controlled scanning electron microscopy (CCSEM) now offer the means to examine thousands of particles per sample in a relatively short period of time (approximately 2 hours vs. 8- to 12-hour examinations per sample using manual microscopy methods), depending on the particles present on the sample.

We hope that this book will spark interest in this ever-expanding discipline and encourage both experienced and novice scientists to revisit GSR interpretation and characterization in forensic casework.

Value of GSR as Trace Evidence

Trace evidence is a type of physical evidence that is often not detectable to the naked eye. Often, some type of magnification or sensitive analytical technique is required to adequately characterize the evidence. GSR evidence is one of the most common and most heavily scrutinized sources of trace evidence examined in violent crime investigations. Understanding GSR and its value as significant trace evidence can aid in the investigation of violent crimes involving firearms. GSR may directly link an otherwise unknown subject to an environment of weapon discharge.

For GSR evidence to be significant, many factors must be considered. GSR is typically found on the hands or clothing of persons who have been in the environment of a discharging firearm, but it may also be found on objects in the vicinity of discharge. It is vital for law enforcement personnel to understand how GSR is created and deposited and how such evidence is collected.

Because the methods of collection and analysis are so varied, it is of utmost importance for those who collect and examine this evidence to be aware of the importance of preserving its potential value. Forensic examiners, evidence collection officers, police officers, EMS personnel, medical professionals, and anyone who comes in contact with suspects and victims following a violent crime must understand the potential existence and significance of GSR evidence. Investigators must corroborate or disprove stories, confirm or contradict suicides, and evaluate scenarios where there are no eye witnesses or other types of investigative leads. For these individuals, education about forensic evidence and training in the area of evidence collection and recognition are essential. It is important to understand the different types of sampling media used in the collection of trace evidence and which type is appropriate for particular crime scene conditions or material to be tested.

The most commonly asked question when a crime involving a firearm has occurred is “Who fired the gun?” (or in the case of a suicide, “Are we sure the victim actually fired the gun?”). These questions can often be addressed simply by taking the time to correctly collect the evidence and properly analyze the samples in the laboratory.

Another common question that arises when GSR is present is “Can you be sure who fired the weapon?” The answer is not always clear. This is why an understanding of how GSR is deposited, how it is collected, its persistence, and the means of analysis are so critical. The examiner must consider all of this information before forming any opinions.

As the reader progresses through this book, it will become apparent that the answers to questions like those asked above are not clear-cut in many cases. What can always be stated with certainty is that detection of GSR means a

person or object was in the vicinity of a discharging firearm at some point in time. If GSR evidence is not properly collected and correctly analyzed, otherwise critical forensic evidence may well remain insignificant or totally undetected. Over the past 40 years, various collection techniques and analytical procedures have been developed and refined. Today, large numbers of samples can be examined quickly to help investigators answer these difficult questions.

Degradation of GSR

Particles of GSR are very durable particles composed of heavy metals that originate from the ammunition components. The inorganic particles that are examined can last in a normal environment indefinitely. Although the actual particles do not degrade under normal conditions, they may not remain on the hands or clothing of a subject over time, because of the physical activity of the subject.

Transfer of GSR

When the firing pin of a firearm strikes the primer cap of the ammunition, a rapid high-temperature burning of the primer mix takes place. This event occurs in a fraction of a second and creates what we refer to as GSR, as well as other things. Particles are deposited on the hands, clothing, and surrounding areas on or near the shooter. The basic principle of trace and transfer evidence is the Locard Exchange Principle.³ This principle states that when two objects come in contact with one another, an exchange of material takes place. A common example of transfer evidence is when two garments come in contact with one another. There may be a transfer of fibers from one garment to the other. Secondary transfer may also occur when trace evidence originally transferred to one object is subsequently deposited on another surface and picked up by a second recipient object, such as a garment.

When one considers GSR evidence, as the firearm is discharged, the particles formed from the combustion products of the ammunition components transfer to the surrounding area. Unlike most transfer evidence, no direct contact is made between the hands and the airborne particulate. The particulate is simply deposited on the hand or surrounding surfaces. Other evidence such as fingerprints from the subject can be transferred to the firearm. Just as secondary transfer occurs in other areas of trace evidence, GSR may be transferred from the hands to clothing or surrounding areas by contact. GSR can be transferred from the backs and palms of the hands to other items simply by rubbing the hands together or transferred from hands into pockets by placing the hands in the pockets.

It is important to understand how evidence such as GSR can be transferred and what significance this type of evidence plays when collected and analyzed by experts who have evaluated the possibilities of these types of situations. To properly evaluate transfer evidence it is important to understand how these particles persist and the environment in which the transfer has occurred. Populations of unique and characteristic GSR particles and other lead-rich particles play an important role when one considers or forms an opinion of what is direct deposition and what is possibly the transfer from hands or clothing due to casual contact. Other variables to consider include environmental conditions, weapon types, ammunition types, caliber, and condition of the weapon. Test firing under static conditions, if the firearm is available, can yield information on particle distribution and deposition in the surrounding environment.

Specific Areas of GSR Deposition

Areas that yield the greatest potential for GSR deposition are investigated by the authors in the plume study video discussed in [Chapter 4](#). Discussion focuses on the areas of highest concentration of GSR deposition — the hands, clothing, and areas surrounding the shooter — using various types of firearms. The information presented in this video can help the collection officer determine what areas offer the greatest potential to recover GSR evidence. A brief summary of key areas of GSR deposition follows.

Hands — The hands are the most common places that GSR is collected from suspected shooters. With firearms such as revolvers, pistols, and automatic weapons, a clear pattern emerges as to the areas of greatest GSR deposition. Collection typically focuses on four regions of the hands (right back, right palm, left back, and left palm).

Face — The face of the shooter can be sampled on occasions when firearms such as rifles and shotguns are used in the shooting. Test firings have shown that large amounts of GSR are deposited in these areas when certain types of weapons are used that cause a condition of blow-back toward the chest, face, and hair.

Clothing — Clothing often preserves GSR evidence even if the hands of the shooter have been cleaned or longer periods of time have elapsed from the time of the shooting and the time of collection. Test firings using numerous types of firearms have resulted in large amounts of GSR deposition on clothing.

Objects in the environment of GSR — Objects that are in the vicinity of GSR may also provide valuable associative evidence that a shooting

occurred in that area. When the plume of the discharging firearm is created, the cloud spreads from all the openings in the firearm and deposits on the areas in the immediate vicinity of the discharge. Examination of tables, chairs, walls, drapes, floors, beds, bedding, items of furniture, and other indoor items may help determine where a shooting took place.

Cars — Drive-by shootings are unfortunately common in today's world. Methods have been developed to collect GSR from the interior and exterior of automobiles involved in these types of crimes. It is not uncommon for the collection officer who employs proper collection methods to recover GSR evidence from automobiles. Seats, seat backs, head rests, dashboards, doors, and headliners are subject to particle extraction using sampling media designed for this application.

Collection of GSR Evidence

Numerous collection techniques are used to prepare samples for GSR analysis. Depending on the technique of choice, protocols are available for the various kinds of testing. For example, the collection officer may need to sample a wide variety of surfaces, such as:

- Skin — hands, face, neck, etc.
- Vehicle surfaces — seat, seat back door, window, dashboard, or headliner on interior or exterior
- Surrounding surfaces — doors, windows, and other body parts
- Clothing — coats, shirts, waistbands, gloves, boots, shoes, hats, etc.
- Any other surfaces in the immediate vicinity of firearm discharge

Although collection procedures vary from agency to agency in the U.S., one procedure that all organizations agree upon is that sterile gloves should be worn when collecting any type of trace evidence. Areas of collection and sampling media are quite different, as observed during a 2-year tour of crime laboratories and agencies in the U.S. The two most widely used collection media types are cotton swabs with a 5% nitric acid solution for Atomic Absorption (AA) and Inductively Coupled Plasma (ICP) and adhesive stubs for scanning electron microscope (SEM) analysis. These devices are commonly used to collect particulate from the hands, face, and other surfaces including clothing, vehicle interiors, and areas in close proximity to a discharged firearm. Swabs are also used to collect particulate from the nostrils and ears of subjects suspected of involvement in the discharge of a firearm.

Collection of samples with 5% nitric acid solution is the method of choice for analytical techniques such as Atomic Absorption (AA), Inductively Coupled Plasma (ICP), or Neutron Activation Analysis (NAA), but it is not a viable sampling medium for scanning electron microscope (SEM) analysis. This is because the nitric acid solution immediately starts to transform the solid particulate into solution, which is retained in the swab media. Virtually all other collection media, such as adhesive stubs, tapes, clothing, skin, gauze, and dry swabs, obtain viable particles for SEM GSR.

Collection areas and number of samples collected, whether by swabbing or dabbing, are not standard throughout the U.S. Some agencies collect hand samples from the back of each hand only. Others collect two samples, combining the back and palm of each hand per sample. Most agencies use four samples: right back, right palm, left back, and left palm. Generally, the areas sampled on the back of the hands cover the index finger, the thumb, and the web area between and behind the thumb and index finger and toward the wrist. Types of adhesive samplers on the market include double-back tape, double-back carbon tape, a multiple-adhesive-layer tape (which requires more extensive sample preparation before the analysis), and the freshly activated adhesive sampler.

Aluminum stubs that are coated with a polymerized adhesive must be coated with a conductive layer or examined in the SEM using variable pressure SEM, which is described in [Chapter 3](#). The SEM equipped with a variable pressure feature requires no special sample preparation and samples are examined in the “as received” condition.

Some samples must be carbon (C) coated because of the nature of the electrons that strike the sample. See [Chapter 3](#) for a complete description of the SEM and its interaction with the sample. The samples are removed and examined in the SEM. The light C coating does not interfere with the analysis of the sample; however, if this process can be eliminated, the possibility of contamination or particle loss from additional handling of the sample during the preparation of the sample for SEM analysis will also be eliminated.

Samples that are collected on double-sided aluminum tape may not be coated prior to sampling because the C-rich background of the tape carries off the electrical charge. This sampler and methods such as variable pressure eliminate the need for C coating.

A multi-adhesive-layer stub, developed by Loren Sugarman in consultation with Dennis Ward, is a collection stub still used by many agencies for GSR collection. The white cap is removed from the sampler and the stub is pressed against a specific area of the hand (e.g., right back, right palm, left back, and left palm) until the tackiness is gone. A slight rotation of the stub while it is pressed against the sampling area will expose new adhesive, allowing prolonged sampling. The cap is replaced and the sample is submitted to

the laboratory for examination. This collection technique requires extensive sample preparation prior to examination in the SEM.

Separate types of sampling media are used for particle extraction from fabric. These types include permanently tacky adhesive stubs, double-sided carbon tape, and adhesive strips for large areas. With an adhesive strip, a large surface area such as a vehicle seat can be concentrated on a small sample (i.e., a 2-in.-by-3-in. strip). A single strip may be used to collect particulate from the entire seat area. To collect particulate from clothing, a shirt for example, one stub is used for the right sleeve, a second stub is used for the right front side of the shirt, a third stub is used for the left shirt sleeve, and a fourth stub is used for the left front side of the shirt. Separate stubs are required for each pant leg. Multiple samplings may be necessary, depending on the coarseness of fibers in the fabric. For example, wool would require more samplings than tightly woven nylon.

The main variables in sample collection are the efficiency of the sample collection officer, the efficiency of the sample collection media, environmental conditions, and the time lapse between the incident being investigated and collection coupled with the physical activity of the subject being examined.

General Crime Scene Considerations

The first and most important consideration in dealing with GSR evidence is the time factor. Studies have shown that, on a living person, GSR persistence decreases with passage of time. *It is very important to collect the evidence as soon as possible after the suspected shooting.* The standard time frame suggested in the literature is 3 to 6 hours after the incident. But in some instances, GSR can persist much longer, especially on clothing and deceased individuals. Improved sampling media may also be a factor in extending the collection time. Protocols vary among U.S. agencies on the effective time period for collecting GSR from the hands of a shooter. Of course, this depends totally on the physical activity of the individual and whether that activity involved the skin or clothing.

Preservation of clothing obtained in the investigation is another important consideration. Do not shake or overly handle clothing of suspects or allow any evidence to be handled extensively or carelessly. Particle loss can be minimized in many cases, and every effort should be made to preserve any evidence that may associate a subject with a suspected shooting. Clothing is a good medium for retaining GSR; unfortunately, its significance in GSR investigations is often underestimated. It may be necessary later to determine which area of clothing was exposed to the GSR “plume.” To aid in this

evaluation, careful preservation of the clothing is important. An understanding of how GSR is deposited in the area of the discharging firearm will also be valuable to the examiner.

Gloves are the best means to ensure that GSR evidence does not come into casual contact with police officers or other law enforcement personnel. A police officer usually does the sample collection. Questions may arise later as to whether the GSR that originated from firing a gun is in fact secondary transfer from the collecting officer. To minimize the likelihood of such confusion and possible accusations, always wear gloves when collecting evidence at a crime scene or from a suspect involved in a crime.

A major misconception is that if you cannot see GSR evidence, it may not be present. Just because you do not see GSR with the naked eye, do not assume that GSR is not there. GSR particles are commonly 1 to 10 microns (μm) in size, although they may range from $<1 \mu\text{m}$ to $>100 \mu\text{m}$ (a micron is 0.00038 in.), so they may not be visible to the naked eye. The appearance of clean hands or unsoiled garments should not lead one to assume that GSR is not present. Conversely, black deposition on the hands or clothing may be from sources other than GSR. You should collect samples even if you do not see what you think GSR should look like. Trace evidence is so named for good reason.

Always document where, when, from whom, and from what areas the GSR was collected from the crime scene. This is critical when the scenario of a crime is formed later in the laboratory. This is especially important when clothing and objects suspected of being in the vicinity of a discharged firearm are examined. GSR evidence has helped corroborate or disprove scenarios in many cases, based solely on whether GSR was present in certain areas of clothing or objects.

Examination of GSR

Any forensic examination begins with properly collected evidence at the crime scene. Knowledge of the various collection techniques and areas of GSR deposition are important for preserving the best evidence for later analysis in the laboratory. Carefully document your crime scene or collection of the evidence. Knowing when and from where the evidence was collected is important for later evaluation of the evidence. It may be necessary to collect GSR at the scene of the crime or shooting. In such cases, the collection officer should understand the various collection methods and collection media available to the immediate law enforcement community.

Laboratory examination is the next step. Samples are prepared, and if necessary, the GSR is examined and evaluated. Various techniques, which we

will discuss later, depend on the efficiency of the collection at the crime scene or the subject. Interpretation of the evidence is not as simple as finding a particle or comparing a concentration of known threshold levels and concluding that a subject may or may not have fired a weapon. An understanding of various factors, such as primer formulations and environmental sources of elements similar to GSR, is critical for interpretation of GSR.

A clear and concise report is an important step in the analysis. The job of the examiner is not finished after the examination is complete; the job is really just beginning. It is the duty of the examiner to help everyone involved in the case to understand what was found and its significance to the case. The effectiveness of the examiner in conveying this information varies and depends largely on the individual's experience and training. Last, and most important, is the duty of the examiner to tell his or her results to the trier of fact. Whether the trier of fact is the judge, jury, or grand jury, the examiner's interpretation of the evidence is critical in helping those who are not experts in the area of GSR understand the pros and cons of what the evidence really means.

Our hope is that this book will lead to a better understanding of GSR and its current role in trace evidence examination.

References

1. Department of Justice, Federal Bureau of Investigation, Uniform Crime Report, Crime in the United States 1997, U.S. Government Printing Office, Washington, D.C., 1997.
2. Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., Final Report on Particle Analysis for Gunshot Residue Detection, ATR-77 (7915)-3, The Aerospace Corporation, El Segundo, CA, 1977.
3. Locard, E., Dust and its analysis, *Police J.*, 1, 177–192, 1928.

Many types of gunshot residue (GSR) analysis are described throughout this book. This chapter deals with the definition of GSR, the mechanism in which GSR is formed and distributed into the environment of the discharged firearm, and current classification schemes for GSR particles. A general description of some common environmental sources of lead (Pb), antimony (Sb), and barium (Ba) is presented.

When the primer mix burns, it escapes from the gun in the form of a vapor or “plume” through any available openings in the weapon. It then solidifies into fine particles and settles onto the hands, clothing, and surfaces in the immediate proximity of the discharged weapon. Immediate proximity varies from weapon to weapon; it is defined by the configuration and condition of the weapon. Other factors that control the effect of particle dispersion include ammunition, be it the manufacturer load or self-loaded cartridges, or the caliber. Air turbulence caused by environmental conditions from either indoor or outdoor currents may influence the flight and dispersion of airborne particulate. Revolvers, semi-automatics with left and right ejection ports, produce different directional plume patterns. These are discussed in [Chapter 4, A Study of Plume Concentrations](#).

The discharging of a firearm initially produces a plume of airborne particulate from the burning primer mix that deposits on any available surface. The particulate of interest is composed of Pb, Ba, and Sb, in varying proportions, and other components of the primer compound. In addition to these inorganic components, many organic components are present in the primer and gunpowder. The combusted material escapes the weapon through the openings as vapors and solidifies as particulate that varies in shape and size from submicron to over 100 microns (μm). GSR analysis determines the presence of these materials on the skin and clothing of subjects and surfaces such as vehicle interiors and exteriors where the particulate may be deposited.

The small, round insert at the bottom of a cartridge casing is called the primer cap. It contains a paste or mixture of primer compound. Three major components in the primer compound play important roles in the formation and identification of GSR:

- The Initiator — lead styphnate, which is set off with a sharp blow, as when the firing pin strikes the primer cap;
- The Oxidizer — barium nitrate, which gives up its oxygen readily; the oxygen is needed to burn the fuel;
- The Fuel — antimony sulfide, which burns at a very high rate; the purpose of this initial burn is to ignite the gunpowder, which propels the bullet.

Particle Formation

The formation of GSR has been studied in detail.¹⁻⁴ The elemental composition of GSR particles may vary depending upon the type of ammunition used, but the method of particle formation is generally accepted in the forensic science community. Understanding particle formation enables the investigator to differentiate GSR particles from environmental sources of Pb, Sb, and Ba.

Because GSR primer particles are formed by the rapid cooling of vaporized Pb, Sb, and Ba following the high-temperature burn of the primer compound, particles of GSR take on characteristic morphologies. Basu¹ describes three classifications of GSR primer particles:

1. Regular spheroids, which are uniformly round particles ranging in size from 1 to 10 μm ;
2. Irregular particles, which are formed by the fusion between smaller and larger particles resulting in nodular spheroids;
3. Particles that form as a layer of lead surrounding an inner core of Ba and Sb.

Particles formed in the interior of the firearm or contained in the cartridge may contain other types of formation. An explanation of particle formation by Wolten and Nesbitt discusses bullet-derived GSR particles.⁴ This is an important issue when one compares the morphology and particle formation of known cartridge casings and uses that information to examine particles found on the hand of a suspected shooter.

Classification of GSR Particles

Are All GSR Particles Spherical?

The Aerospace Report⁵ states that “70–100 percent of the **unique** particles in a sample of GSR residue are spheroidal.” The morphology and the relative brightness of the heavy metal particles enabled early examiners to detect possible unique particles and distinguish them from background material.

In the early days of manual microscopy, an examiner spent long hours on one sample looking for small, round, bright white particles, staring at field after field of magnifications, hundreds of particles per sample. These examiners did exceptional work, considering the methods they had at their disposal. With the advent of computer-controlled scanning electron microscopy (CCSEM) an automated system now detects all potential GSR particles containing any heavy metals characteristics. The examiner can then relocate to the specific particle, already shown to have possible chemistry consistent with GSR. The examiner no longer has to manually search, since all particles spheroidal and amorphous are examined for chemistry unique to and characteristic of GSR. The alleviation of the manual approach enables the examiner to focus only on certain particles consistent with GSR.

With today’s automated GSR systems, examiners look at thousands of particles in 10% of the time previously required. The CCSEM does all the time-consuming work of screening for potential GSR particles. As a result, only those particles with significant elemental compositions are reviewed manually to classify particles.

If an examiner were to examine all amorphous particles that exhibited heavy metal characteristics, the process would be too time-consuming for samples with heavy concentrations of metal particles, not all of which are unique to GSR composition. Using CCSEM techniques, a high number of amorphous particles are detected that contain the elemental composition unique to GSR. The following study evaluates the amount and the particulate morphology of GSR particles as detected using the CCSEM/Energy Dispersive Spectroscopy (EDS) system.

Shape and Texture of GSR

GSR particles are generally spherical in shape; however, collections taken immediately after test firings yield particles with varying morphologies and surface textures. Many particles show overall cracking characteristics with edges that are straight or jagged to partially rounded. When sampling is performed immediately after weapon discharge, and before any physical activity takes place, a true representation of particle size and distribution,

shape, and morphology can be observed and analyzed. When populations of particles are examined under these conditions, it is readily apparent that GSR particles are not limited to spherical, bright particles less than 10 μm in diameter. In fact, the distribution of particles from samples taken from various primer/ammunition manufacturers have been analyzed with similar results. Large populations of irregularly shaped GSR particles are present in all the primer residues.

As the various GSR particles collected from the shooter's hands immediately following the discharge of a firearm show (see [Chapter 5](#)), a wide range of particle size distributions and shape factors govern GSR particles. In many large particles, the surface cracking shows the potential breakup into smaller particles with straight edges. Even the cracking through spherical particles reveals the potential for irregular shapes with combinations of straight- and rounded-edge particles. With physical activity, i.e., rubbing and clenching of hands, these particles may be lost or broken into several sections and redistributed over the skin and clothing of the shooter. Certainly many spherical particles remain intact and in agglomerated form; however, the irregularly shaped particles with the unique composition of GSR should not be disregarded.

Morphologies of Known GSR Particles from Test Firings

Two revolvers, a Colt Trooper .356 and a S&W 1917-.45, and two semi-automatic weapons, a S&W MOD 39-9mm and a Colt 1911A1-.45, were test fired in multiple firings using ammunition from four manufacturers (CCI, FED, REM, and WIN). A sampling of the data from the CCSEM analyses, evaluating the unique particles only for shapes of these particles (spheroidal vs. irregularly shaped), revealed the following information.

1. For the revolvers, 74% of the particles analyzed were spheroidal and 26% were irregularly shaped.
2. For the semi-automatic weapons, 59% of the particles were spheroidal and 41% were irregularly shaped.
3. In addition, by size, in the revolvers, 65% of the particles were $<3 \mu\text{m}$, 28% were 3 to 6 μm , 5% were 6 to 10 μm , and 2% were $>10 \mu\text{m}$.
4. For the semi-automatic weapons, 48% of the particles were $<3 \mu\text{m}$ in size, 29% were 3 to 6 μm , 9% were 6 to 10 μm , and 14% were $>10 \mu\text{m}$.

The results of this limited evaluation of test-firing particles indicate that when an analyst has relocated and observed countless flagged particles with unique and characteristic chemistry, the experienced eye of an examiner is capable of evaluating the possible source of spherical and irregularly shaped particles. In many cases, the examiner can visually interpret agglomerations

of combined spheroidal and broken particles, layered particles and particles attached to other masses, and single particles that could never be mistaken as spheres. Irregularly shaped particles that exist in significant numbers with chemistries consistent with GSR have been routinely characterized in laboratories. The determination that GSR particles are only spherical or possess some degree of three-dimensional roundness has been disproved in many test cases utilizing CCSEM. Whenever these particles are taken from a pristine surface that has only been exposed to the discharge of a firearm, there can be no argument as to their source. This is a significant difference from most earlier methods of GSR particle characterization.

Uniqueness of Composition

When Pb, Sb, and Ba combine into a single particle, it is defined as being unique to GSR, since there is no other known source of this compound. Each individual element or other combinations of these elements with high temperature features are classified as characteristic of GSR.

Terminology

Since the Aerospace Report was prepared in 1977, many advances in GSR have occurred, and several interpretations of GSR evidence have been modified by numerous examiners. Environmental source traces from vehicle brakes and various types of fireworks have shown the potential to resemble GSR. Although this is a concern of many analysts, GSR, like other sciences, is an ever-evolving field, and examiners must stay current with research to achieve the greatest understanding in evaluating GSR evidence. Newer methods of examination have been developed since the days of manual SEM and were utilized to classify particles in the 1970s literature.⁵ Methods such as CCSEM are now the norm, and Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) and organic methods of analysis show promise in the field. Some laboratories have even proposed multisampling methods such as bulk and SEM, areas which were never evaluated in the 1977 Aerospace Report.

We suggest a new way of describing GSR particulate be evaluated, which considers these recent techniques, similar environmental particulate, and other developments in the ever-changing world of GSR examination.

Based on the current literature⁶ and our present research in the area of environmental particulate with elemental compositions similar to GSR, our laboratory designates only Pb-Sb-Ba as unique GSR particles.

Unique:	Pb-Sb-Ba
Characteristics:	Pb-Sb
	Pb-Ba
	Sb-Ba
	Sb
	Ba
	Ba-Ca-Si (with trace S)

[Note: Minor concentrations of the following elements may be present in gunshot residue: Si, Ca, Al, Cu, Fe, S, P, Zn (only with Cu), Ni, K, Cl.]

As many examiners in the field of GSR examination have described, the total evaluation of all the particulate present should be used to determine if the potential particles are in fact consistent with GSR. GSR is a valuable type of physical evidence, but its significance must be understood and explained in a way that considers all potential variables of GSR. Particle morphology must be looked at more closely because of CCSEM, which was not available when many standards for examining GSR were formulated. Controlled test firings have shown large populations of irregularly shaped particles, simply because of the newer methods of testing. In the past, examiners looked for bright spherical particles, because such particles had the greatest potential for having an elemental composition consistent with GSR. Today's methods determine the elemental composition first, then review the particles for morphology. This has led to detection of a higher number of irregularly shaped particles. These test firings are void of environmental contaminants and show a high concentration of irregular GSR. These are important considerations in evaluating an automated GSR examination.

Environmental Considerations in GSR Examination

Elements present in the composition of GSR are also found in many forms and combinations in the environment. The metallic elements Pb, Ba, and Sb are used extensively in numerous applications in the commercial world. However, these three elements, in the form of lead styphnate, barium nitrate, and antimony sulfide, have only been combined in a single application for one product: the mix in the primer cap of a cartridge casing. Studies in progress involve new formulations of certain fireworks products that may have mixes similar to primer ammunition. Thus far, none of the samples tested has yielded particles that would be classified as unique to GSR. Particulate from certain brake-lining manufacturers' products are also being examined by researchers in the U.S. and abroad, although the product formulation has been changed to meet environmental regulations. New products and formulation changes must be constantly monitored when Pb, Sb, and Ba are part of the product

fabrication. In any case, overall populations of particulate must be carefully examined for elemental tags accompanying the particles when classifying particles and sources of the material in question. Examples of some of the chief commercial or industrial uses of Pb, Sb, and Ba are as follows.⁷

Lead (Pb) — melting point 327°C/621°F

Storage batteries, solder, bearing metals, gasoline, bullets, shot, paint, shielding sheet, cable coverings, glass, matches, dyes, and pyrotechnics

Antimony (Sb) — melting point 440°C/824°F

Hardening alloys (lead base, etc.), babbitt metals, paint pigment, safety matches, bearings, pewter utensils, brake pads, and pyrotechnics

Barium (Ba) — melting point 850°C/1562°F

Deoxidizing alloys of tin, copper (Cu), Pb, and zinc (Zn), radiography, in heat-treating baths for tempering of steel, in laser crystals, dyes, inks, leather tanning, photographic chemicals, insecticides, paper products, and pyrotechnics

These elements are used in the applications listed above and numerous others, but the end result of these applications does not produce particulate that would be identified as GSR. The decisive factors in the identification of GSR are the specific composition, morphology, and chemistry of the particle, as well as the characterization of the overall population of particles detected on the same sample.

References

1. Basu, S., Formation of gunshot residues, *J. Forensic Sci.*, 27, 72–91, 1982.
2. Zeichner, A., Levin, D., and Dvorachek, M., Gunshot residues formed by using ammunitions that have mercury fulminate based primers, *J. Forensic Sci.*, 37, 1567–1573, 1992.
3. Zeichner, A., Levin, N., and Springer, E., Gunshot residue particles formed by using different types of ammunition in the same firearm, *J. Forensic Sci.*, 36, 1020–1026, 1991.
4. Wolten, G. M. and Nesbitt, R. S., On the mechanism of gunshot residue particle formation, *J. Forensic Sci.*, 25 (3), 533–545, 1980.
5. Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., Equipment Systems Improvement Program — Final Report on Particle Analysis for Gunshot Residue Detection, ATR-77 (7915)-3, The Aerospace Corporation, El Segundo, CA, Sept. 1977.

6. Mosher, P. V., McVicar, M. J., Randall, E. D., and Su, E. H., Gunshot residue — similar particles produce fireworks, *Can. Soc. Forensic Sci. J.*, 31 (2), 157–168, 1998.
7. Brady, G. S. and Clauser, H. R., *Materials Handbook*, 13th ed., McGraw-Hill, New York, 1991.

Methods for Examination of Gunshot Residue

3

Principal Techniques

The techniques for the detection of gunshot residue (GSR) are well documented and have evolved over the last several decades. Early methods such as nitrate tests were replaced by methods such as Neutron Activation Analysis (NAA), Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS), and Graphite Furnace Atomic Absorption Spectroscopy (GFAAS). Since the publication of the Aerospace Report in 1977, much emphasis has been placed on the use of Scanning Electron Microscopy/Energy Dispersive Spectroscopy (SEM/EDS) because of its ability to obtain both morphological information and the elemental composition of the particles. Recently, SEM/EDS systems have become automated, making automated GSR by computer-controlled SEM the method of choice for most laboratories conducting these analyses.

Currently, the principal techniques used for the detection of GSR are Flameless Atomic Absorption Spectrometry (FAAS) and ICP — both bulk analytical techniques — and particle analysis by SEM/EDS. Capillary electrophoresis (CE) is another method of analysis currently being investigated.

Scanning Electron Microscopy/ Energy Dispersive Spectroscopy (SEM/EDS)

Bulk analysis can detect trace amounts of lead (Pb), barium (Ba), and antimony (Sb) in a sample, down into the nanogram range. The detection of these elements is therefore suggestive of GSR. However, this only proves that Pb, Ba, and Sb were present on the subject without correlating them to a single source; i.e., in principle, each element could have been generated independently. These results, by nature, are nonspecific.

SEM analysis, in contrast, analyzes on a particle-by-particle basis, thus retaining the individual feature characteristics lost in a bulk analysis. The SEM can tie the presence of Pb, Ba, and Sb to a single particle. Furthermore, the SEM is capable of detecting down to a single, micron-ranged GSR particle on an entire sample, which is picogram-level analytical sensitivity. Clearly, SEM is the preferred technique from the standpoint of specificity and positive identification of trace levels of GSR.¹

Theory of the SEM

Since the measuring device has been constructed by the observer ... we have to remember that what we observe is not nature in itself but nature exposed to our method of questioning.

— Werner Karl Heisenberg

A variety of instruments can be used to assist the eye in observing shape, size, and composition. Different types of instruments offer different advantages in GSR analysis. Some instruments allow us to see objects that are too small or too distant to be observed with the naked eye. Other instruments analyze energy in more detail to better constrain or even identify the composition.

For example, optical microscopes use polarized light to identify minerals, whereas optical telescopes and other instruments have spectrometers that measure the wavelengths of emission or absorption bands in specific regions of the electromagnetic spectrum to identify the elements or compounds responsible for them. When analyzing materials, it is best to use whatever instrument or instruments are best suited to the material and the question at hand. The SEM must be included in the list of instruments considered. The SEM mimics what our eye sees and how our brain interprets an image.

Features of the SEM

Several features of the SEM make it useful in many forensic studies, and especially in GSR analysis, including magnification, imaging, composition, and automation. These features are outlined below; they will be discussed in detail in the remaining text.

Although the SEM is noted for its high magnification capabilities, it also functions at the low magnification necessary for some applications. For example, in GSR analyses the sample is commonly scanned at a magnification of 100×. Compared to an optical microscope, the depth of focus is considerably greater in the SEM and the entire particle remains in focus.

The SEM can be equipped to image in two modes. The secondary electron mode yields an image with a three-dimensional appearance. The back-scattered electron mode yields an image that relates brightness to composition and is thus useful for general phase differentiation.

The SEM is usually equipped with an X-ray analyzer and an EDS, also referred to as EDX (for energy dispersive analysis of X-rays), which yields information about the elemental composition of the material being analyzed. As opposed to bulk chemical analytical procedures, the chemistry obtained by the SEM is of a very small volume, say a few cubic micrometers (μm). In this way, elements associated with one another in a chemical compound are identified. A bulk chemical analysis of a GSR sample may reveal that Pb and Sb are present at nanogram levels, but it cannot determine if these elements are independent or associated in a compound. The SEM analysis will identify specific phases.

The modern computer-driven SEM acquires the images in a digital format. Programs have been written to move the stage and beam in an automated fashion. Image analysis procedures can be applied to field images to identify “features,” those objects that are either brighter or darker than the surrounding matrix. The combination of backscattered electron imaging, EDS, and computer control allows samples to be analyzed automatically, and is commonly referred to as computer-controlled scanning electron microscopy (CCSEM).

How the SEM Works

As mentioned above, objects can be observed and described in terms of how they interact with energy. Therefore, we will divide our discussion into three components: the source of energy, the interaction of energy and the specimen, and detecting the effects of that interaction. In the case of the SEM, we are concerned with the column which has a source of electrons (gun) and a series of lenses (optics) that produces a beam; how this beam of electrons interacts with the specimen; and how the resulting emissions (electrons and X-rays) are detected and displayed. These three components are described in the following three sections. Some of the points that the analyst should know when acquiring quality data are listed.

The Column

The first task is to produce an intense and highly localized source of electrons. This can be accomplished in several ways. One of the most common ways is to use a heated wire filament that is bent at an acute angle to make a shaped point from which the electrons are emitted. The emitter is maintained at a negative potential of up to several thousand volts, so that electrons that are “boiled off” (Figure 3.1A) the heated tip are accelerated toward the anode,

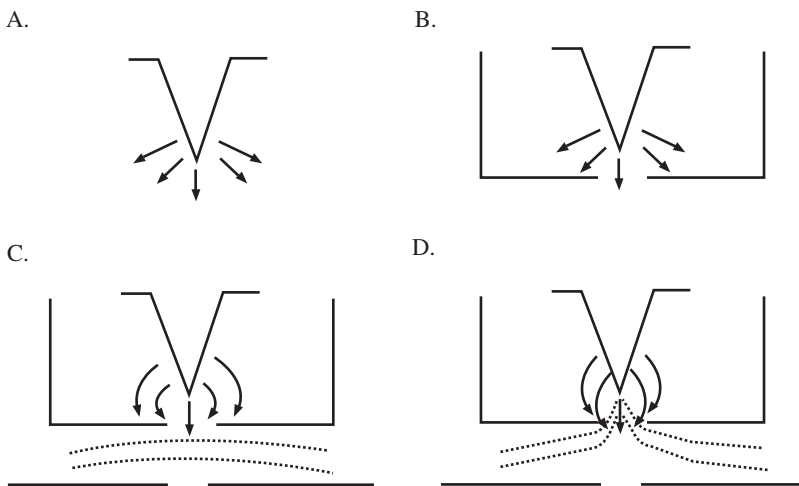


Figure 3.1 Gun cap configuration. (A) Electrons given off by heated filament are scattered in all directions. (B) Electrons directed toward the column by a Wehnelt. (C) Electrons attracted toward the column by a positive charge on the anode. (D) Bias on the Wehnelt deforms the anode field for more efficient collection of electrons.

which has a central hole allowing the electrons to enter the column. A bias voltage is applied to the grid structure that encloses the filament, so as to focus the beam to a small point, called the “crossover.” The rest of the column lenses further demagnify this already small beam.

Below the filament is a Wehnelt (“vay-nalt”), part of a lens that focuses the electrons (Figure 3.1B). Electrons passing through the Wehnelt are accelerated into the column. The anode is positive relative to the cathode but only because the Wehnelt is operated at a negative voltage (Figure 3.1C). The positive charge, referred to as the accelerating voltage, ranges from perhaps 1 kilo-electron volt (KeV) to 50 KeV, depending on the particular instrument. A small negative bias voltage placed on the Wehnelt deforms the anode field to help draw the electrons into the column (Figure 3.1D).

To turn on the beam, the filament drive current is increased to heat the filament and to emit more and more electrons, similar to the way the dimmer switch of a light fixture functions. At a certain point, additional filament heating produces no appreciable increase in electron emission. At this point, the filament is said to be saturated, and the beam is energetic and stable.

The accelerated electrons pass through a hole in the anode into the SEM column. The column consists of a central “tube” surrounded by magnetic lenses that produce a small focused beam, circular in cross section. This beam passes through several spray apertures to reduce the amount of scatter in the beam and ultimately through the final aperture. The diameter of this aperture

is important in that a larger aperture allows more beam electrons to illuminate the sample, resulting in a brighter image. However, the angle of convergence of individual electrons in the beam will be larger in a wide aperture, and this will reduce the depth of focus. A balance between these two factors is achieved by the selection of the diameter of the final aperture. The depth of focus is related to the angle of the “cone” of electrons, which in turn is determined by the aperture of the electron microscope. The aperture on the SEM is very small, generally on the order of 100 μm , which results in a depth of field on the order of over 100 μm at 100 \times and several tens of μm at 500 \times . In the case of GSR where particle size is generally 10 μm or smaller and these are analyzed at about 100 \times , the entire particle is in simultaneous focus. Such is not the case with optical microscopes.

The optics of SEM can change over time as small amounts of contamination accumulate on the conductive elements of the column, particularly on the final and spray apertures. In addition to general environmental contaminants, specimens placed in the vacuum of a sample chamber may not be inert and outgassing may occur. The product of such contamination allows charges to accumulate on surfaces and deforms the beam so that it is no longer circular in cross section but “astigmatic” instead. The image resulting from this condition will be lower in resolution. SEMs are equipped with a special set of crossed focusing elements (stigmators), which can be applied independently to make the beam circular. The effects are first noticeable at higher magnifications but a severe astigmatism will eventually affect images down to the 100 to 500 \times range.

Column Issues

We can summarize the issues with regard to the gun and column that affect the quality of the results as follows:

1. The filament should be aligned in its structure.
2. The filament should be properly saturated.
3. The beam should be aligned with respect to the anode.
4. The proper bias should be placed on the Wehnelt.
5. Imaging should be assessed for astigmatism and corrected by applying the stigmator or cleaning the column as needed.

Beam/Sample Interaction

We recall from chemistry that material is composed of atoms and that atoms are composed of a central nucleus of protons and neutrons with orbiting electrons in one or more “shells.” In the order of increasing distance from the nucleus, these shells are referred to as K, L, M, etc. When a beam of electrons (called primary electrons) hits the specimen, the electrons penetrate

a distance directly proportional to the beam energy and inversely proportional to the specimen density. As the primary electrons interact with specimen atoms, the energy is dissipated within an interaction volume. From 20 to 30% of the primary electrons collide with specimen atoms, rebound elastically with little energy loss, and are scattered out of the specimen. The reemergent beam electrons are known as backscattered electrons. When the primary electron beam hits the specimen, atoms may be ionized by the forced emission of electrons. The specimen electrons emitted from the sample are referred to as secondary electrons.

When secondary electrons are emitted from an atom, they create “holes” in the electronic configuration. These holes will be filled by the collapse of electrons from any higher order shell. This process is called an “electron jump.” Higher order electrons are less tightly bound to the atom than those in inner shells, and when they “jump” to an inner vacancy their excess energy is released as characteristic X-rays, so called because the properties of the X-ray (energy and wavelength) are characteristic of the element from which it was derived.

As one goes up the periodic table of the elements, the electron configuration becomes more complex. Specimen electrons can be emitted from any of the shells, and the resulting holes can be filled by electrons from any higher order shell. A naming convention has been established for the various emitted X-rays. The first character refers to the shell in which the electron hole was created and into which an electron jumps. This is named after the shell, e.g., K, L, M, N. The second character refers to the number of shells involved in the jump: alpha (α) for one shell, beta (β) for two shells, gamma (γ) for three shells. This convention is illustrated schematically in [Figure 3.2²](#) and as labeled spectral peaks in [Figure 3.3](#).

Remember that the SEM works by bombarding the specimen with a beam of electrons. Some portion of these electrons emerges from the specimen as backscattered electrons, but others do not. Because the specimen is in a vacuum the electrons are not easily bled off into the chamber. Therefore, the specimen should be a conductor of electrons. Because many materials of interest are natural conductors, no special considerations are needed other than to ensure that there is a continuous path to ground through the specimen holder. However, other materials of interest are not natural conductors. In these samples, electrons will build up to the point that they make the image negatively charged and thus render the image unusable.

For nonconductors, the general solution is to give the specimen a thin coating of conductive material, such as gold (Au) (or Au and palladium (Pa)) or carbon (C). Each has advantages. Au and Pa are very good conductors and the resulting images may be better than those coated with C. However, these relatively heavy elements interfere with the beam electrons, sample

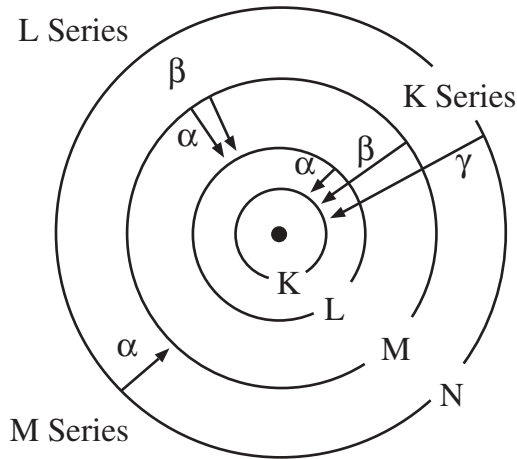


Figure 3.2 Naming convention for X-rays generated by specific electron jumps (redrawn from Postek 1980).

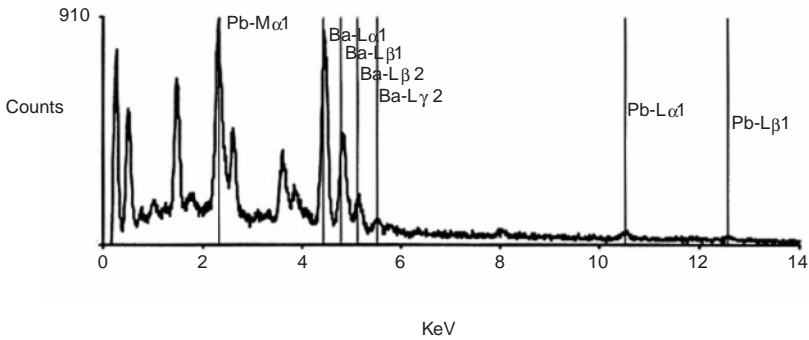


Figure 3.3 EDS spectra of “unique” gunshot residue particle showing the identification of multiple peaks for Pb and Ba.

electrons, and emitted specimen X-rays, and contribute Au/Pa X-rays that cannot be differentiated from X-rays generated from Au or Pa in the specimen. C does not absorb as much energy and generates fewer interfering peaks, but the problem of differentiating coating vs. specimen C remains.

A more recent approach to the elimination of charging effects is to maintain some atmosphere in the specimen chamber. Of course, the filament needs to remain in a vacuum, but this is not required of the specimen. To be sure, the atmosphere in the specimen chamber does interfere with the electrons. However, with an appropriate amount of gas in the chamber, charging dissipates, and these interactions turn out to be relatively minor. Perfectly adequate analysis of nonconductive samples can be achieved in many cases using such a setup. Most manufacturers provide this option,

which is referred to as variable pressure operation, among other names. This capability is taken to a higher level of pressure in an environmental-type SEM that allows greater freedom in the amount and nature of the atmosphere in the specimen chamber.

Another advantage of the variable pressure operation is that nonconducting specimens do not have to be altered by the coating process, thus allowing larger samples (an entire glove, for example) to be accommodated.

The following issues regarding beam/sample interaction should be addressed:

1. To avoid charging effects, be sure the sample has a continuous path to ground.
2. The word “point” is often casually used to refer to the intersection of the beam with the specimen surface. Keep in mind that this region is in fact a small volume. The distribution of X-rays obtained from a single phase consisting of two elements may be identical to the distribution of X-rays obtained from two closely spaced single element phases.
3. X-rays will not be generated unless there is sufficient energy in the electron beam to excite the elements of interest (“over-voltage”). Make sure you have sufficient accelerating voltage to identify the elements of interest.

Detectors

The SEM is usually equipped to detect electrons generated by two different processes for imaging, secondary and backscattered, and with X-rays for compositional analysis.

Electron detectors. The electrons are emitted from the sample surface in all directions, but in varying proportions. Consider the case of electrons emitted from a flat sample with an incident electron beam perpendicular to the surface (Figure 3.4A). For both secondary electrons (SEs) and backscattered electrons (BEs), the greatest number of electrons will be emitted perpendicular to that surface. The number of electrons generated at various angles is represented by the length and direction of the arrows in Figure 3.4. The length of the arrow of any direction is defined by a circle, as shown in Figure 3.4A.

Where the surface is at an angle, the SEs and the BEs behave differently. The greatest number of SEs will again come off in a direction normal to the surface (again defined by a circle in Figure 3.4B). However, the greatest number of BEs will come off at an angle, as if reflected, as illustrated by the ellipse in Figure 3.4B.

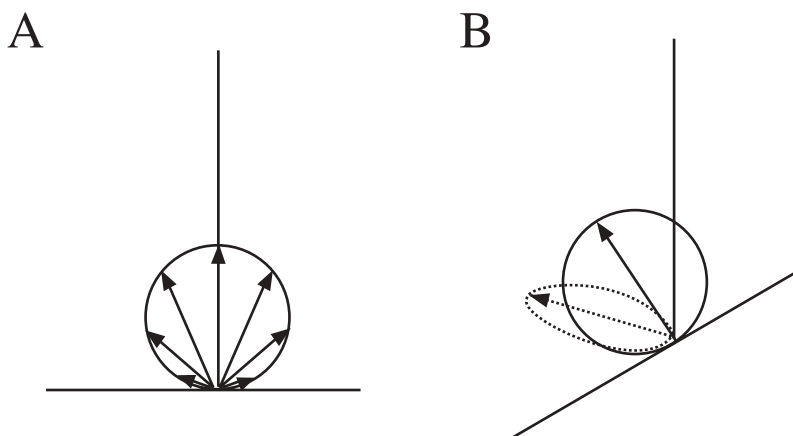


Figure 3.4 Relationship between incident beam and direction of emitted electrons from a perpendicular surface (A) and from an angled surface (B).

The BEs are “reflected” beam electrons and are highly energetic. The SEs are generated in the specimen and are relatively low energy.

The most commonly used SE detector is the Everhart-Thornley (ET) detector, named for its inventors. The ET detector consists of a collector, scintillator, light pipe, and photomultiplier. The SEs are relatively low in energy and emerge from the specimen radiating in all directions. They are attracted to the collector of the ET detector by its small positive charge; that is, the path of SEs can be curved in the direction of the detector. A scintillator element is provided with a large positive charge that accelerates the electrons toward it. The electrons emit light when they crash into the scintillator, and this light is carried by the light pipe out of the specimen chamber to an external photomultiplier tube where the light energy is converted to an electric current. The SE detector can also detect and process BEs. The high energy BEs are not attracted to the detector by the small positive bias, so only those electrons that were directed to the detector upon emission from the specimen will be collected. The ET detector is a rather inefficient detector for the BEs.

Two BE detectors are widely used: scintillator and solid state. A scintillator type BE detector is similar to the ET detector described above but larger in size and positioned so that more BEs strike it. A solid-state type BE detector consists of a semiconductor “diode.” When struck by a BE, the semiconductor produces an electron-hole pair, the components of which migrate in opposite directions under the influence of a small applied field. These moving charges produce an electrical current. No positive bias is applied to this detector since small charges will not attract the fast-moving BEs. In fact, some instruments have a small negative charge applied to the

detector. This charge is not sufficient to repel the BEs, but it will repel the SEs, resulting in a purer BE signal.

Imaging

Up to this point, we have discussed events that occur when a stationary electron beam is directed at a specimen. This process may be useful for determining the composition at a “point” (read “small volume”), but no image can be produced in this mode. To produce an image, scan coils in the column cause the beam to raster or scan over an area of the specimen. This is the scanning in the SEM. The electrons (either SEs or BEs) are detected at known beam positions in that raster and the image is reconstructed on a display monitor. Regions where electrons were detected in large numbers are displayed as relatively bright compared to regions where electrons were detected in small numbers. Older instruments usually displayed the images as analog output on a phosphor CRT and hard copy images were obtained through photography. Newer instruments display digital images that can be saved and/or printed as computer files.

Magnification is related to the sizes of the raster compared to the size of the display. When the beam raster is large, the image displayed on the screen shows relatively low magnification. When the beam raster is small, the image displayed on the screen shows relatively high magnification. This results in the magnification of the SEM.

The two different kinds of detectors produce different kinds of information when collecting images. Because the path of SEs can be somewhat curved, shadows are considerably softened. This results in an image with a three-dimensional aspect. The path of BEs is not curved, so the resulting image appears “flatter.” However, the major advantage of images produced in the backscattered electron (BSE) mode is that phases can be discriminated. Because like charges repel, electrons “bounce off” the electrons surrounding the specimen atoms and scatter back out of the specimen. The number of BEs scattered back out of the specimen is proportional to the number of SEs surrounding an atomic nucleus. The higher the atomic number, the greater the number of specimen electrons, and the greater the likelihood that beam electrons will strike specimen electrons, resulting in a greater number of electrons that will be scattered back and a brighter image.

The bottom line here is that the higher the atomic number of an object, the brighter the object will appear. Although this information usually cannot be used directly for phase identification, it can be used for phase differentiation and screening. For example, small particles of quartz and feldspar derived from soil will be relatively dark compared to Pb, Ba, and Sb particles derived from GSR. When looking at a sample for GSR, examination would

be in the BSE mode, thus passing up the “haystack” of darker particles while singling out the GSR “needles.”

Imaging Considerations

The SEM infers the size of a displayed object based on a calibration curve for a specific magnification. The operator should check the size calibration periodically by comparing the measured size of an object to the known size of that object. A variety of size standards are available for this purpose. Keep in mind that the column should be degaussed prior to performing any size measurements. (See the discussion of working distance, below.)

X-Ray Detectors

The SEM is commonly equipped with an EDS that determines elemental composition. An EDS unit is a very sophisticated device that accurately measures the very miniscule amount of ionization current produced when an X-ray is absorbed in a silicon sensing element (silicon diode). To accomplish this feat, the diode must be maintained at a very low temperature reservoir of liquid nitrogen (at -196°F) and very specialized circuitry must be employed.

The EDS detector processes a single X-ray at a time, and while doing so it is not available to process additional X-rays. The actual time that the detector is not processing data and is available for analysis is referred to as live time. Conversely, the time involved in processing is referred to as dead time. For efficient analysis, it is recommended that dead time reside in the approximate range of 20 to 40%. At lower dead time, the processor is idle much of the time, waiting for the next X-ray. At higher dead times, the X-rays come in so quickly that there is a high probability that a second event will interfere with the measurement of a first event. This phenomenon, referred to as “flooding” or “swamping” the detector, should be avoided.

Despite continued improvement in detector technology, the signal-to-noise ratio imposed by the fundamental physics of detector operation is still such that the measurement of the amount of energy carried by an X-ray photon has some degree of error. Because this error is random, the resulting plot of successive X-rays from a single element is represented by a peak of perhaps 100 to 150 eV wide rather than a single discrete value. Because these peaks are broad, there may be overlap among peaks of various elements, which sometimes makes element identification problematic. This problem is alleviated somewhat by the fact that most overlaps do not involve the exact peak location, that peaks of overlapping elements are not necessarily exactly the same shape, and that heavier elements commonly have additional peaks not present in the lighter elements.

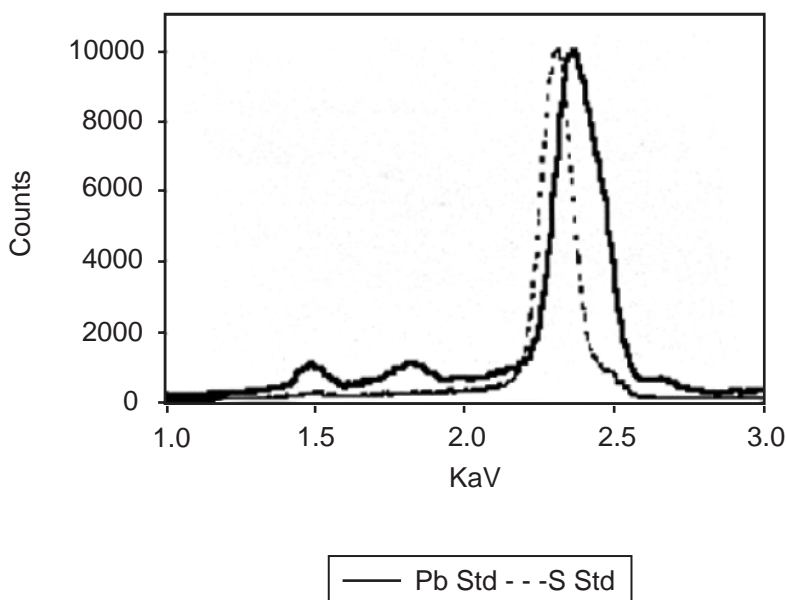


Figure 3.5 Peak overlaps of Pb and S.

An example of a peak overlap of interest in GSR is Pb and sulfur (S). Both of these elements have their major peaks at about 2.3 KeV, the α peak for S and the α peak for Pb. As Figure 3.5 shows, the peak shapes are not identical. The Pb peak is somewhat asymmetrical because it is indeed comprised of two closely spaced peaks. In addition, the Pb has α and β peaks at about 10.5 and 12.6 KeV, respectively.

In cases of peak overlap, the analyst must take care to identify the element responsible for that peak, e.g., long EDS acquisition time, expanding the scales for detailed inspection, and comparison to known elemental standards. Sophisticated software is also available to deconvolve such overlaps.

Another solution to resolve ambiguous calls based on EDS is to use the wavelength dispersive spectrometer (WDS). Wavelength is inversely related to energy and is thus just another measure of the same property. Wavelength can be more accurately measured using a WDS than can energy using an EDS, and the resulting peaks are very narrow. The peaks that display considerable overlap in EDS are observed as discrete peaks in WDS. However, an entire EDS spectrum for all elements excited in the beam interaction volume can be processed simultaneously and quickly (a matter of seconds). WDS analysis proceeds on an element-by-element basis and requires many minutes of operator time for each element. WDS units are also considerably more expensive to purchase and maintain. Some SEMs have optional WDS capabilities. This detector is common on electron microprobes.

X-Ray Artifacts

Other effects associated with X-ray detectors may lead to incorrect conclusions concerning the composition of a specimen.

We mentioned that the EDS detector processes a single X-ray photon at a time. Two photons may enter the detector simultaneously so that the detector observes them as a single event. The energy is then interpreted as the sum of the energy of the two photons combined, and the resulting peak is referred to as a “sum peak.” This does not occur often enough to be a problem except when great numbers of X-rays enter the detector. This can be avoided by reducing the spot size or increasing the working distance. Modern processors do a better job excluding multiple photons than do older processors, but sum peaks may still occur.

The EDS detector is comprised primarily of silicon and is bombarded by energetic X-rays. On occasion a silicon X-ray can be excited in the detector material, thereby reducing the energy detected by the amount of energy it took to liberate the silicon X-ray (e.g., 1.74 KeV). This shows up as a peak 1.74 KeV lower than the element peak, and it is referred to as an “escape peak.”

The specimen chamber is a closed system with high-energy electrons and X-rays ricocheting about. These particles can stimulate the emission of X-rays from the construction material of the chamber, most commonly iron (Fe). On occasion, the geometry is such that these X-rays can be generated and detected in sufficient quantities to result in an observable peak.

Working Distance

Working distance (defined as the distance between the bottom of the pole piece and the specimen surface) is important with respect to image quality and X-ray detection. The strength of the signal is a function of distance (the inverse square law) and the detector geometry. The best working distance depends on the instrument being used, but in general the best distance for imaging is not necessarily the best distance for EDS. For example, the best imaging distance may be around 10 mm or less, whereas the best EDS working distance may be around 20 mm. A compromise distance should be chosen depending on the needs of the analysis.

The SEM cannot directly measure the working distance to the specimen but must infer it from the amount of current needed to drive the final lens so that it focuses the beam on the surface. However, as the lens current is changed repeatedly, residual magnetism may build up in the lens material. This distorts the computations of working distance and thus produces an incorrect magnification value. This residual magnetism can be eliminated by a process known as “degaussing,” which should be done on a regular basis, and particularly before the magnification is calibrated.

EDS Considerations

Check the EDS calibration on a regular basis, say, once a month. This EDS calibration check is generally performed by acquiring a spectrum of copper and identifying the energy of the center of the K and L peaks. These peaks should occur at 8.04 and 0.93 KeV, respectively. It is recommended that the system should be recalibrated if the peak positions are off by ± 0.03 KeV (30eV).

Know the possible peak interferences for each identified element. When these elements are observed, care should be taken to ensure their correct identification.

Bear in mind the significance of beam-sample-detector geometry, since X-rays can be shadowed by sample shape. When analyzing particles (as in GSR) the composition of features on the surface facing away from the detector cannot be determined.

Automated SEM Analysis

Computer-controlled scanning electron microscopy (CCSEM), otherwise known as “automated SEM,” is the most efficient method employed in the analysis of GSR.³⁻⁵ Such a system can rapidly screen thousands of particles and store the composition, size, images, and location coordinates of potential GSR particles. To give such data evidentiary value, the flagged particles must be relocated and individually examined to confirm identity.

Automated SEM coupled with energy dispersive X-ray analysis is the current method of choice for detection and identification of GSR. The major advantage of using SEM/EDS in the examination of GSR is the ability to obtain morphological characteristics and elemental compositions from individual particles in a short time, without examiner fatigue. With SEM/EDS, the examiner may have the option of examining evidence using variable pressure. This technique allows the examiner to utilize SEM on various surfaces and items of evidence without elaborate sample preparation.

The SEM analysis on a particle-by-particle basis retains the individual feature characteristics and can relate the presence of Pb, Sb, and Ba to a single particle. When the instrument detects particles that contain Pb, Sb, or Ba, it flags the particles as potential GSR and stores images, composition, and coordinate data for relocation and confirmation by manual microscopy after the automated analysis is completed. A summary sheet is printed with stored images and spectral data for relocation and confirmation applications. Particle size distributions can be acquired with an off-line program. Following the automated analysis, each particle flagged as potential GSR should be relocated and its morphology and composition manually characterized.

In most cases, GSR samples collected on adhesive SEM stubs can be placed directly into the SEM for analysis. Some collections are performed on multiple-adhesive-layer stubs, which require preparation by dissolving the adhesive and redepositing the particulate on an SEM stub.

In some fields of investigation, the analyst can prepare the samples so there is a uniform distribution over the entire sample area, such as with controlled experiments. With GSR casework, we do not have this luxury. GSR samples must be analyzed in the “as collected” condition.

An ASTM method suggests that if the analyst were required to identify a minimum of five GSR particles for a positive result and 45% of the stub had been analyzed with no particles detected, this would indicate with 95% certainty that any further analysis would be unlikely to detect the particles required for a positive result. If 60% of the stub were analyzed with no GSR particles detected, the certainty factor that the result would not be positive would increase to 99%.

Contrary to this theory, there have been samples in cases where the last 15% or the first 15 to 20% of samples has yielded GSR particles and the balance of the sample area had no particles unique to GSR. This is because the cited statistical criterion assumes random distribution of material. However, GSR sample collection is anything but uniform; often, nonuniform distribution of GSR can occur. Sample collection over the hands or other areas using adhesive stubs does not provide uniform distributions of particulate as in the ideal sample preparation techniques used under laboratory conditions. For example, when a collection officer dabs the hands of a subject, it may not always be possible for the officer to place the stub directly down and flat on the sampling area. Sometimes the stub will make contact with an edge produced by movement of the subject’s hand or the placement of the stub may be affected by the various areas of different concentrations. In any case, nonuniform distributions are issues that the GSR analyst must deal with under real-life sample collection conditions.

Since GSR concentrations may be segregated on any area of the sample surface, the orientation of the sample placement on the holder in the SEM becomes a critical factor in the analysis of any given stub. The analysis must be allowed to be continuous if no GSR particles have been detected at any point in the analysis to guarantee 100% certainty of the presence or lack of GSR on the sample.

Variable Pressure SEM

The variable pressure SEM has proved an extremely valuable tool that eliminates the need for C coating and various sample preparation techniques

previously used to ready the samples for analysis. Particle loss and possible contamination during preparation are critical parameters in the analysis of GSR, and extreme care must be exercised in the handling of these samples.

In a conventional SEM, some type of sample preparation is often needed to provide a sample that is compatible with the electron beam of the microscope. If the sample is nonconductive, there is no path for electrons to travel away from the sample at the point of beam contact or impact. This causes a charge buildup that blooms bright in that localized region and washes out the features of the image. The sample may require a coating or film of C or Au to eliminate the charging effect. Samples may have outgassing problems, caused by oil, grease, or moisture. Some materials outgas naturally under vacuum due to porosity. Other materials may have high vapor pressure properties. If a sample is outgassing, it may require several hours of evacuation before the electron beam of the conventional SEM can be turned on. Some samples are evacuated for long periods of time in a separate chamber to remove oil vapor or moisture before they can be placed in the microscope for examination. Numerous materials require intricate sample preparation techniques when the examiner uses a conventional SEM.

A SEM with a variable pressure feature can eliminate the need for sample preparation on most materials and, at the same time, minimize the possibility of contamination during the process. As discussed earlier in this chapter, a vacuum system is necessary for operation of the SEM. For high vacuum integrity, generation, acceleration, and control of the electron beam must be maintained. However, it is possible to maintain the high vacuum required in the gun assembly, thus creating an efficient condition for beam generation and then, through a series of restrictive orifices, to isolate the sample chamber and regulate the pressure at an elevated level. This bleeding of gas into the sample chamber supplies additional gas molecules, which are ionized by the action of the beam, causing dissipation of surface charge, as noted by Schamber.⁶

Various samples that were once considered virtually impossible or extremely difficult to prepare and examine due to charging and outgassing are now being examined with no preparation at all. Saturated oil filters, concrete, fabric, and many other materials had to be rotated and tilted for coating procedures or prepumped for hours and placed in and taken out of the microscope for additional preparation to obtain satisfactory imaging and compositional information necessary for analysis. GSR samples collected on a nonconductive adhesive substrate can be examined routinely at a sample chamber pressure of 0.2 torr and a working distance of 16 mm. When one considers the time saved in sample preparation for just one set of samples, then multiplies that by the case load of a busy laboratory, one understands why a variable pressure SEM is a welcome, time-saving addition to GSR analysis.

This is an extremely useful technique when one considers the number and diversity of samples that may develop a charge in the SEM. In the application of GSR, this method has increased the efficiency of analysis by eliminating the need to coat SEM stubs and tape lifts as well as providing the examiner the added luxury of placing a piece of fabric “as is” into the chamber for analysis. Because a sample may not be coated, particles that may be lost in the preparation process are not lost or disturbed.

In the following case examples, a variety of sampling media were used to collect GSR. Samples were collected from the hands of subjects using double-backed carbon tape SEM stub samplers, activated adhesive stub samplers, and 3M™ 465 double-sided tape SEM stub samplers. Permanently tacky SEM stub and strip samplers were used to extract particles from clothing and vehicle interiors.

A variable pressure SEM (PERSONAL SEM™) was used in the appropriate vacuum mode, depending on the type of sampling media used in each case (e.g., carbon tape samplers usually do not require variable pressure). Many of the samples in this study would be difficult to examine in the standard SEM without coating. Fabric, for instance, requires extensive rotating and tilting during the coating procedures.

The following case studies illustrate the importance of variable pressure SEM in the analysis of GSR. In these instances, variable pressure SEM reduced the possibility of particle loss and minimized contamination issues.

Case Study 1

Suede and woven-fiber gloves (Figure 3.6) were worn during the test firing of a Colt .45 revolver to illustrate the appearance of GSR particles trapped in the weave of the fabric. The glove worn on the shooting hand was placed in the SEM and scanned manually to locate the area with the heaviest population of GSR particles (Figure 3.7). The area was sectioned from the glove and placed flat on the SEM stage for examination (Figure 3.8). This procedure is followed when the weapon is known and circumstances in the case suggest that particular items of clothing may have been exposed to GSR. After the weapon has been identified, it is possible to isolate areas of high potential GSR.

A standard SEM GSR collection kit with double-sided tape-type samplers was analyzed using a SEM in the variable pressure mode. Carbon coating was not used prior to the analysis. Two hundred and thirty (230) particles were flagged as potential GSR particles on the right back hand sample. Eight particles were classified as characteristic of GSR; four particles were classified as unique to GSR with a composition of Pb-Ba-Sb. The total analysis time was 59 min, 43 s. Figure 3.9 illustrates typical particles confirmed as unique to GSR.

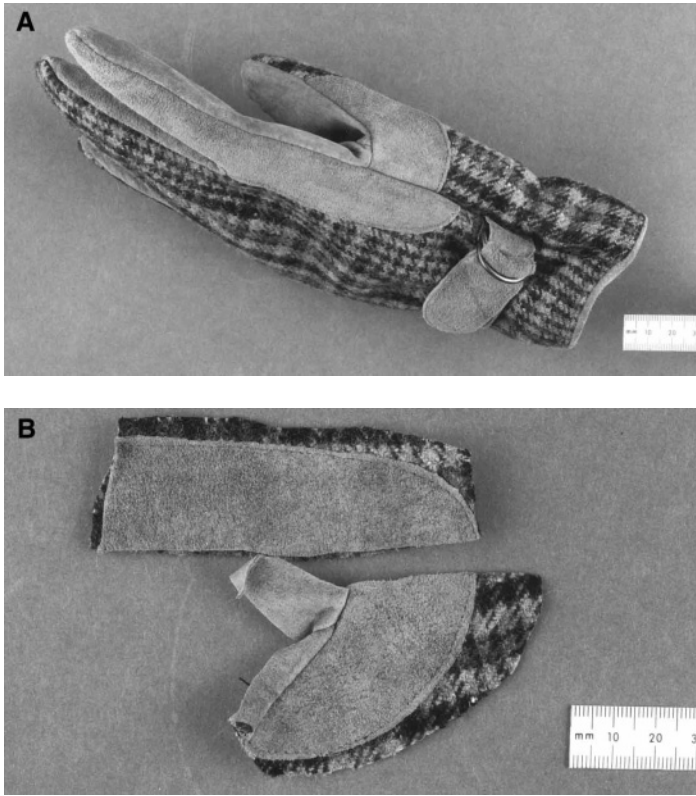


Figure 3.6 Suede and woven-fiber glove worn during test firing (A) and after being sectioned for analysis (B).

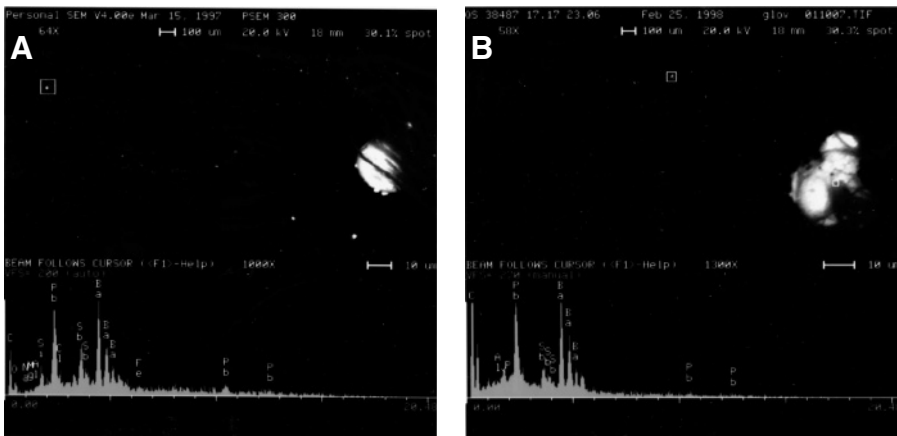


Figure 3.7 BE image (A) and EDS spectrum (B) of particles recovered from areas within the weave of the glove. Both particles exhibit compositions unique to GSR (Pb, Ba, Sb).

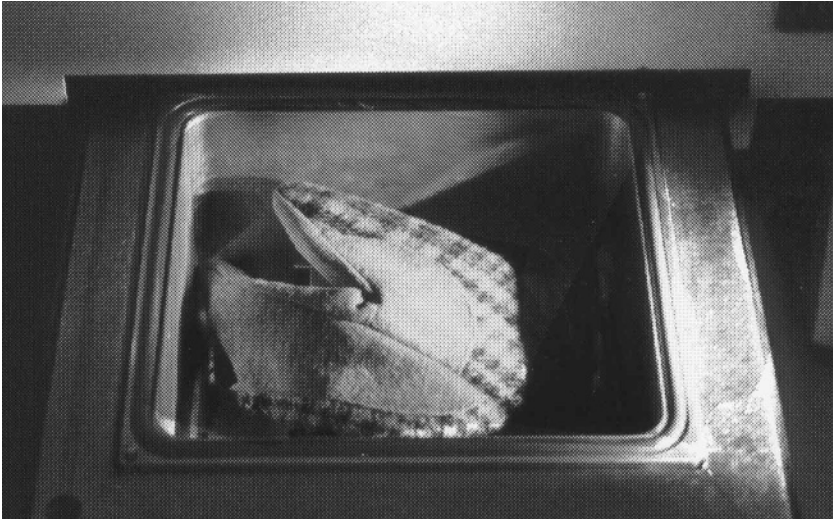


Figure 3.8 Insertion of the sectioned glove into the SEM chamber for examination by variable pressure SEM.

Case Study 2

Permanently tacky SEM adhesive stubs were used to extract particles from the clothing of a subject. A variable pressure SEM was used to detect particles unique to GSR on samples from the shirt, pants, hat, and shoe of the suspected shooter. The SEM stubs were placed directly into the SEM without any additional sample preparation, e.g., C coating. Nine samples were analyzed in approximately 11 hours. [Figure 3.10](#) illustrates unique GSR particles extracted from the clothing.

Case Study 3

Permanently tacky adhesive strips were used to extract particles from a vehicle headliner, seat, and door panel for an examination involving the discharge of a firearm inside the vehicle. The samples were examined in the “as received” condition using variable pressure SEM, thus eliminating the need for C coating. Following CCSEM analysis several particles were classified as unique to GSR. [Figure 3.11](#) illustrates a representative unique GSR particle as confirmed by the relocation and “live” confirmation of a particle that was flagged as potential GSR particles during the automated analysis.

Case Study 4

Particles were extracted from the shirt, inside diameter of the waistband of a pair of pants, and shoes of the subject and examined in the variable pressure

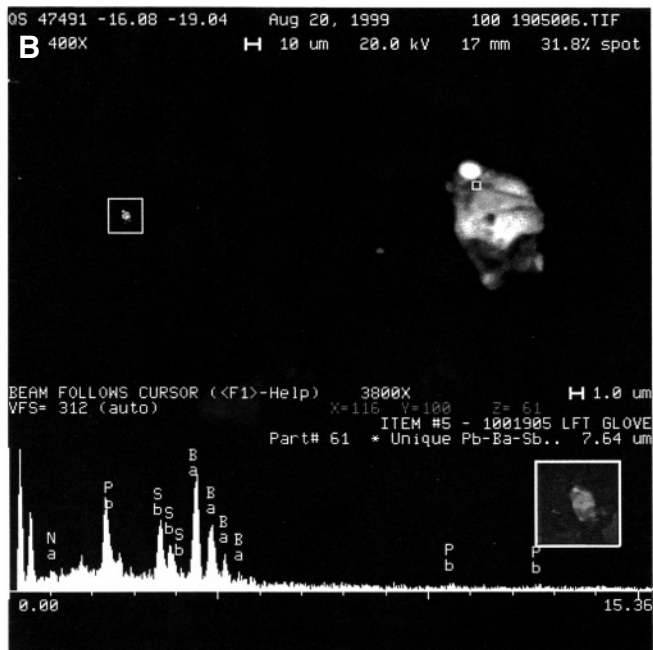
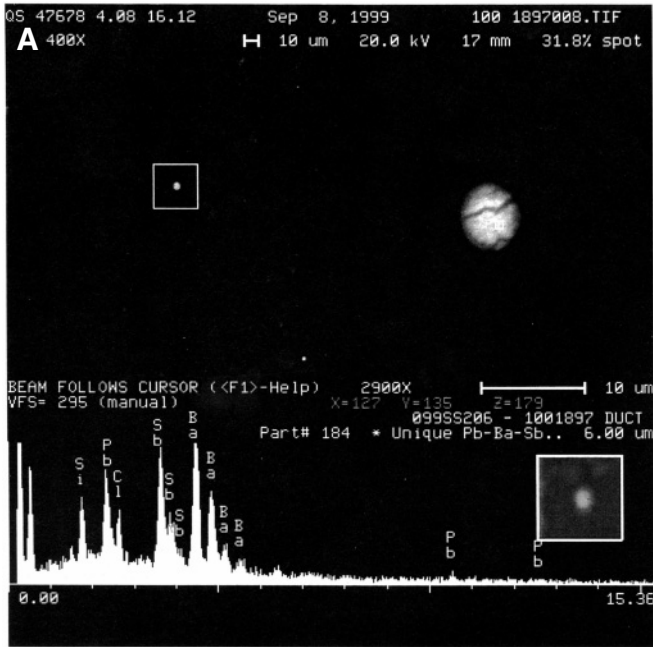


Figure 3.9 Typical particles recovered from GSR collection kit analyzed in the variable pressure mode.

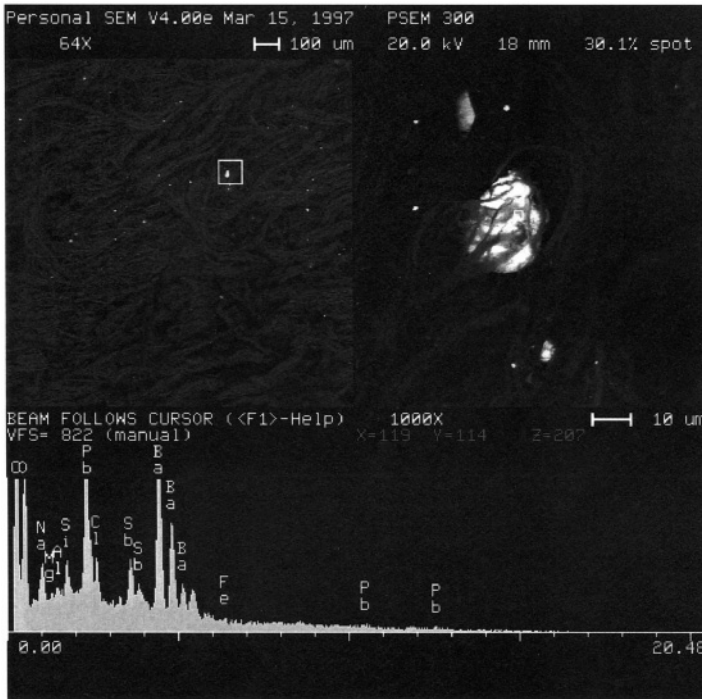


Figure 3.10 Unique GSR particle extracted from the shirt of a suspected shooter and examined using variable pressure SEM.

mode of the SEM. Unique and characteristic particles were classified and confirmed by real-time relocation. Typical unique GSR particles are shown in [Figure 3.12](#).

Discussion of Variable Pressure SEM

Variable pressure SEM coupled with CCSEM has made a tremendous impact on laboratories with high volumes of trace evidence samples. The time saved in sample preparation alone is invaluable to the analyst. The likelihood of particle loss and possible contamination due to preparation techniques are greatly reduced. These conditions would appear to relieve the caseload and backlog problems that plague most crime laboratories; however, consequences of rapid and efficient analyses appear to produce an increase in caseload, as reported in a survey of crime laboratories conducted by *USA Today*.⁷ New detectors have further increased the speed and accuracy of GSR analysis. Perhaps there is light at the end of the tunnel as technology improves to meet the needs of the seemingly never-ending quest of the forensic scientist for the perfect system.

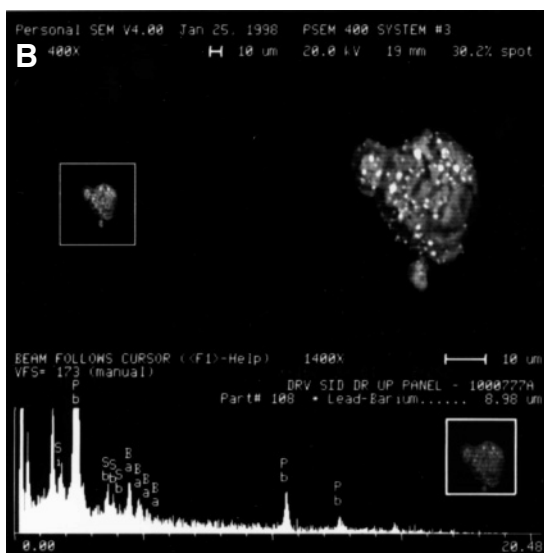


Figure 3.11 Photograph of the extraction procedure from vehicle door panel (A) and the confirmed “unique” GSR particle (B).

Other Methods of Analysis

Although automated SEM/EDS analysis is the method of choice for most laboratories conducting GSR analysis, other methods may be utilized and have proved valuable in detection and characterization of GSR. The following are general descriptions of other past and current techniques used by laboratory personnel to examine GSR.

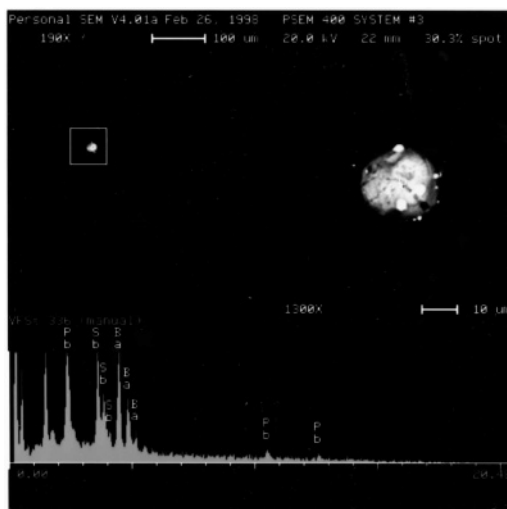


Figure 3.12 Typical particles unique to GSR extracted from the inside of a pair of pants and analyzed using variable pressure SEM.

Color Tests

Color tests are commonly used by examiners to determine range of firing estimations.⁸⁻¹⁰ Although firing range determination is not covered in this text, it is important to understand the various methods and chemical tests used to indicate that residue from a discharging firearm may be present. In many instances, SEM is not available or may not provide information needed by examiners. For instance, if an examiner knows that a shot has been fired into a fabric and must determine the distribution of the expelled residue on the clothing, one of the various chemical tests may aid in the investigation. It must be pointed out that the following methods are only presumptive in nature and are not specific to GSR.

The Paraffin Cast or Dermal Nitrate Test relied on the color reaction of the nitrate recovered on the sampling surface with diphenylamine reagent. A warm paraffin was poured on the hands of a suspected shooter and allowed to cool. The cast was pulled off of the hands and the adhering residue was subjected to diphenylamine reagent. Although this method gave a positive reaction for nitrates, many other substances also gave positive results for this test, thus making this method an unreliable indicator of GSR.

The Sodium Rhodizonate test is a chemical test for the presence of Pb. The main benefits of this test are the ease of performance and low cost. The Walker test is a chemical test for the presence of nitrates in unburned or partially burned propellants. Like the aforementioned tests, this test gives investigators valuable information about the range at which a weapon was

fired or whether the suspected hole was an entrance or an exit hole, assuming the shot occurred in close proximity to the garment. The modified Greiss test is used to detect nitrites and the Diphenylamine Test is used to detect the presence of nitrates and nitrites. Other color tests used to determine firearm distance include the Marshall and Tewari tests, both of which test for nitrites. The Lunge reagent is a chemical used to indicate the presence of nitrocellulose.

Bulk Methods of Analysis

Graphite Furnace Atomic Absorption Spectroscopy (GFAAS)

Graphite Furnace Atomic Absorption (GFAAS) is a bulk method of analysis used in the analysis of inorganic materials in primer residue, namely, Ba and Sb. After sample preparation, the Ba and Sb are in a liquid state. A graphite tubular furnace chamber temperature is raised by a programmed electrical power source. A fixed volume of the sample is analyzed during furnace atomization.

The sample is subjected to a drying stage, an ashing stage, and an atomization stage in which free atoms are generated. The absorption signal measured during the atomization stage is proportional to the amount of analyte element in the sample. These amounts are typically reported in g/ml. Sample preparation methods for GFAAS have been studied in detail by Koons et al.¹¹⁻¹³

Neutron Activation Analysis (NAA)

Neutron Activation Analysis (NAA) is a bulk method of analysis in which the inorganic components of primer residue can be determined. This method of analysis for the characterization of GSR has been described in the literature.¹⁴ Sample preparation involves irradiation of the sample using neutron bombardment, which forms radioactive isotopes by combining neutrons with elements contained within the sample. The neutron-induced radioisotopes emit X-rays which are characteristic of the individual elements, and the trace level amounts are determined by the peak radiation intensities. This technique, although accurate, is not widely used due to cost, lack of availability of reactors to examiners in the field, and time required to complete the analysis.

Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS)

Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) is a bulk method of analysis used to measure trace amounts of Pb, Ba, and Sb in primer residue samples.¹⁵ It operates by coupling an argon plasma to a radio frequency (RF) electromagnetic field at atmospheric pressure. Samples are usually introduced in liquid form. Detection limits are usually in the parts-per-billion level.

Capillary Electrophoresis (CE)

Micellar Electrokinetic Capillary Electrophoresis (CE) is a bulk method of analysis that is designed to separate and detect organic and explosive material components.¹⁶ It is a multiple-wavelength analysis technique that generates ultraviolet spectral profiles and selective wavelength monitoring.

References

1. DeGaetano, D. and Siegel, J. A., Survey of gunshot residue analysis in forensic science laboratory, *J. Forensic Sci.*, 35, 1087–1095, 1990.
2. Postek, M. T., Howard, K. S., Johnson, A. H., and McMichael, K. L., *Scanning Electron Microscopy*, Ladd Research Industries, 1980, 73.
3. Tillman, W. L., Automated gunshot residue particle search and characterization, *J. Forensic Sci.*, 32(1), 62–71, 1987.
4. White, R. S. and Owens, A. D., Automation of gunshot residue detection and analysis by scanning electron microscopy/energy dispersive X-ray analysis (SEM/EDX), *J. Forensic Sci.*, 32, 1595–1603, 1987.
5. Germani, M. S., Evaluation of instrumental parameters for automated scanning electron microscopy gunshot residue particle analysis, *J. Forensic Sci.*, 36, 331–342, 1991.
6. Schamber, F. H., *Basic SEM: An Introduction to the Personal SEM*, Trafford, PA, RJ Lee Instruments Limited, 1997.
7. *USA Today*, Crime Laboratory Survey, September 20, 21, 1987.
8. Saferstein, R., *Forensic Science Handbook*, Vol. 1, Regents/Prentice-Hall, Englewood Cliffs, NJ, 588–589, 1982.
9. Harrison, G., Firearms Discharge Residues, *J. Forensic Sci.*, 188, April, 1959.
10. Owens, M. and George, W., Gunshot residue examinations: modification in the application of the sequence of chemical tests, *AFTE J.*, 23(4), 1991.
11. Koons, R. D., Havekost, D. G., and Peters, C. A., Analysis of gunshot primer residue collection swabs using flameless atomic absorption spectrophotometry: a re-examination of extraction and instrument procedures, *J. Forensic Sci.*, 32, 846–865, 1987.
12. Koons, R. D., Havekost D. G., and Peters, C. A., Analysis of gunshot primer residue collection swabs using flameless atomic absorption spectrophotometry and inductively coupled plasmaatomic emission spectrometry effects of a modified extraction procedure and storage of standards, *J. Forensic Sci.*, 34, 218–221, 1989.
13. Koons, R. D., Flameless atomic absorption spectrophotometric determination of antimony and barium in gunshot residue collection swabs: a collaborative study, *Crime Lab Digest*, 20, 19–23, 1993.

14. Krishnan, S. S., Gillespie, K. A., and Anderson, E. J., Rapid detection of firearm discharge residues by atomic absorption and neutron activation analysis, *J. Forensic Sci.*, 16, 144–151, 1971.
15. Koons, R. D., Analysis of gunshot primer residue collection swabs by inductively couple plasma-mass spectrometry, *J. Forensic Sci.*, 43(4), 748–754, 1998.
16. MacCrehan, W. A., Smith, K. D., and Rowe, W. F., Sampling protocols for the detection of smokeless powder residues using capillary electrophoresis, *J. Forensic Sci.*, 43(1):124–199, 1998.

A Study of Plume Concentrations

4

The Plume Study

The initial purpose of the plume study undertaken by RJ Lee Group, Inc. in August 1994 was to provide U.S. law enforcement agencies with a better perception of gunshot residue (GSR) plume development and potential sampling areas for a number of firearms. A “plume” is the gaseous formation that exits all openings of a firearm following the discharge of a round of ammunition.

The plume study examined the plume concentrations from the discharge of weapons most commonly used in the U.S. The representative sample of various types of firearms chosen encompassed the general characteristics of most available firearms. This study was undertaken following extensive evaluation of sampling protocols practiced by various law enforcement agencies in the U.S. The objective of the study was to show the area of highest potential for the collection of GSR under normal firing conditions.

In the study, a closed indoor firing range was used to eliminate air drafts, thus allowing for the observation of plume direction under static conditions. A black backdrop was placed parallel to the firing directions. Front and rear flood lighting enhanced the observations of plume development from the weapon discharge. A 31-min video tape recorded the plume development and areas of heaviest potential GSR concentrations. Test firings were recorded with a high-speed motion analyzer at 500 to 1000 frames per second, revealing a slow-motion plume development for each weapon.

Results of the plume study were determined by visual observations of the video and computer-controlled scanning electron microscope (CCSEM) (RJ Lee Instruments PSEM) analysis of GSR particles collected using adhesive samplers. For handguns, a single shooter stood with one arm fully extended, to show the best view of the weapon to be tested. Right- or left-handed test firings were determined by the action of the firearm, whichever yielded the best observation of any given weapon, i.e., ejection port position, cartridge ejection, and plume concentration. Shooters fired rifles and shotguns in the

standing or sitting positions, depending on weapon recoil, to keep the firearm in the total field of view of the camera.

It is our hope that the information gained from this study will aid in the understanding and collection of GSR evidence.

Colt 1911



Figure 4.1 Colt 1911 (45 ACP).

History: This semi-automatic pistol was the standard U.S. military pistol from 1911 to 1985, when it was supplanted by the Beretta 92-F. This firearm is still used by the U.S. National Guard. The Colt Model 1911 is currently manufactured by Colt and the three other U.S. manufacturers; it has been copied extensively. It is produced in China by Norinco and imported into the U.S. for sale. Copies of this firearm are currently made in Spain, Argentina, and Mexico.

Description: This pistol has a barrel length of 5 in. and a 7-shot clip capacity. The ejection port is 90° and ejects to the right, open at the top.

Objective: To show the effect of a larger caliber pistol vs. the 9 mm.

Area of Highest Plume Concentration: The heaviest plume concentrations for the Colt 1911 were behind the second knuckle of the trigger finger extending back, and the web of the hand to the wrist area.

Walther P-38



Figure 4.2 Walther P-38 (9mm Luger).

History: This semi-automatic pistol was the first double-action military pistol. It was adopted by the German army in 1938 and by the German land forces after World War II. This pistol is still currently available.

Description: This pistol has a barrel length of 5 in. and an 8-shot clip capacity. The ejection port is open a full 180°.

Objective: To show the effect of a 180° ejection port vs. a 90° ejection port typified in the Colt Model 1911 A1.

Area of Highest Plume Concentration: From the front of the fingers, extending to the back of the web.

Victoria 1911

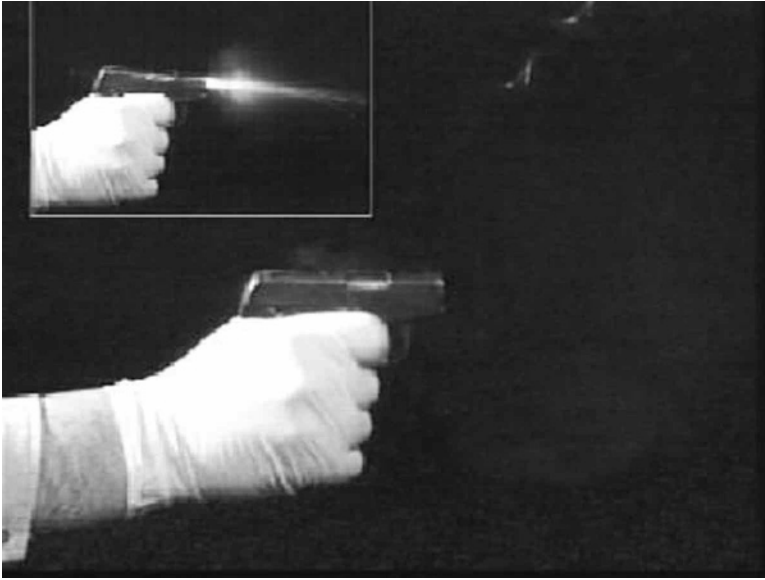


Figure 4.3 Victoria 1911 (25 ACP).

History: This semi-automatic pistol is the Spanish copy of the Colt/Browning Model 1908. Several hundred variations of this representation of the Colt/Browning models exist.

Description: This pistol has a barrel length of 2 in. and a 6-shot clip capacity. The ejection port is 90° to the right.

Objective: To show the effect of the 25 ACP cartridge.

Area of Highest Plume Concentration: From the front of the fingers to the web.

Colt Trooper

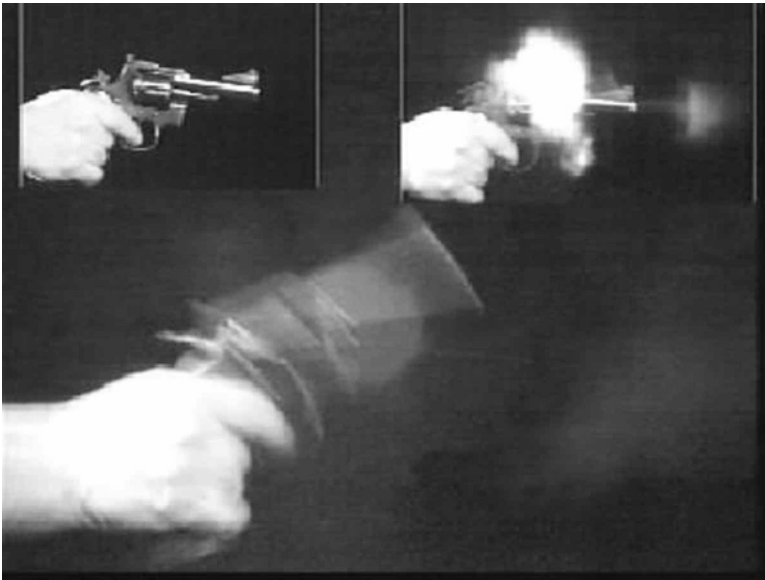


Figure 4.4 Colt Trooper (357 Magnum).

History: This weapon typifies the current Colt revolvers in use today.

Description: This revolver has a barrel length of 4 in. and a 6-shot cylinder. The firing pin is separate from the hammer, an inertia-type built into the frame.

Objective: To illustrate the effect of a revolver with standard barrel length.

Area of Highest Plume Concentration: The discharge engulfs the entire hand, with a slight wisp over the web on multiple firings.

Smith & Wesson Model 36



Figure 4.5 Smith & Wesson Model 36 (38 Special).

History: This weapon, known as the “Chief’s Special,” is a very common concealed weapon.

Description: This revolver has a barrel length of 2 in. and is built on the J frame. It has a 5-shot cylinder. The firing pin is on the hammer.

Objective: To illustrate the effect of a short-barrel revolver.

Area of Highest Plume Concentration: A wide plume develops over the fingers and hand, with the heaviest concentration at the web and a heavy blow-back to the wrist.

Smith & Wesson Model 34



Figure 4.6 Smith & Wesson Model 34 (22 Long Rifle).

History: This gun is built on the same frame as the Smith & Wesson Model 36 (38 Special).

Description: This weapon is also built on the J frame. It has a 2-in. barrel and a 6-shot cylinder. The firing pin is separate from the hammer, inertia type, built into the frame.

Objective: To show the effect of the .22 vs. a .38 with a 2-in. barrel.

Area of Highest Plume Concentration: From the web to the wrist and the entire trigger finger to the front of the fingers.

Ruger Mark II Automatic



Figure 4.7 Ruger Mark II Automatic (.22 Long Rifle).

History: This is a very common .22 semi-automatic firearm.

Description: This semi-automatic pistol has a $4\frac{3}{4}$ -in. barrel, a 9-shot clip, and a 90° ejection port to the right.

Objective: To show the effect of the .22 semi-automatic pistol.

Area of Highest Plume Concentration: A heavy concentration between the second and third knuckles of the trigger finger and a light concentration between the third knuckle and the web.

Colt 1903



Figure 4.8 Colt 1903 (.32 ACP).

History: This gun is typical of the class of semi-automatic “pocket pistols.”

Description: This firearm has a $3\frac{3}{4}$ -in. barrel with an 8-shot clip. The ejection port is 90° to the right.

Objective: To show the effect of the .32 ACP cartridge in a “pocket pistol.”

Area of Highest Plume Concentration: From the second knuckle of the trigger finger, extending to the area behind the web.

Duo 25



Figure 4.9 Duo 25 (25 ACP).

History: This semi-automatic pistol was made in Czechoslovakia after World War II. It is also typical of the Colt/Browning Model 1908 design.

Description: It has a 2-in. barrel with a 6-shot clip. The ejection port is 90° to the right.

Objective: To illustrate the similarities of two .25 ACPs from different manufacturers.

Area of Highest Plume Concentration: A light plume from the second knuckle of the trigger finger and heavier as it moves forward to the front of the fingers.

Smith & Wesson 39



Figure 4.10 Smith & Wesson 39 (9mm Luger).

History: This semi-automatic pistol is typical of the many Smith & Wesson models currently available, e.g., 59, 439, 459, 639, 659, etc. This was the first double-action pistol manufactured in U.S. It is a very common pistol in the U.S. It is used by the Illinois State Police.

Description: This is a semi-automatic pistol with a 4-in. barrel and an 8-shot clip. It has a 90° ejection port to the right.

Objective: To illustrate the differences among the Smith & Wesson, Browning, and Walther P38 models.

Area of Highest Plume Concentration: An even distribution from the first knuckle of the trigger finger, extending back to the web.

Browning Hi Power



Figure 4.11 Browning Hi Power (9mm Luger).

History: This model was the last pistol designed by Browning. It introduced the double-stagger, high-capacity magazine. It is the current military pistol used in Canada, Great Britain, and other countries. This pistol is still made and copied in Hungary and Argentina.

Description: This pistol has a 13-shot clip and a 4 $\frac{5}{8}$ -in. barrel length. The ejection port is 90° to the right.

Objective: To illustrate the differences among the Smith & Wesson, Browning, and Walther P38 models.

Area of Highest Concentration: The highest visible concentration is from the third knuckle of the trigger finger, back over the web of the base of the thumb.

Smith & Wesson 1917

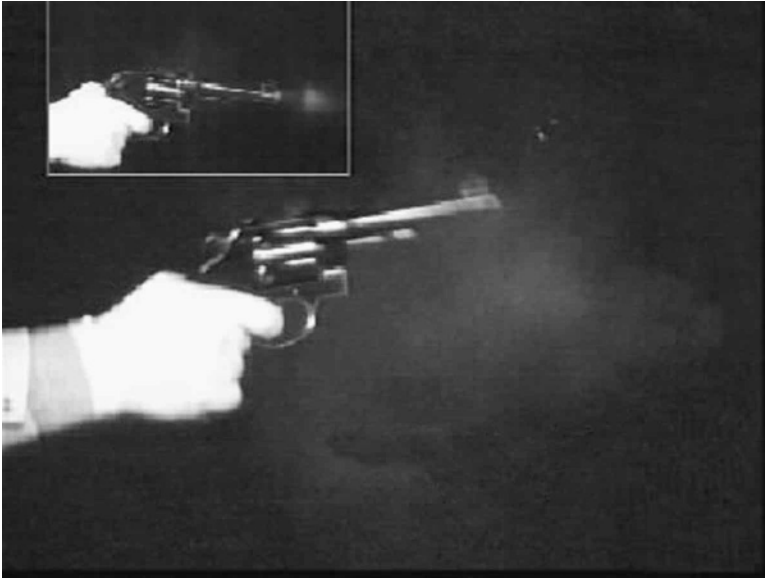


Figure 4.12 Smith & Wesson 1917 (45 ACP).

History: This was a World War I U.S. substitute pistol because there were not enough Colt Model 1911 auto-loaders available. A commercial model was produced by S&W into the 1960s.

Description: This revolver has a 6-shot cylinder and a 6-in. barrel length. The firing pin is integral with the hammer and is built on the N frame.

Objective: To show the effect of a larger caliber bullet in a double-action revolver.

Area of Highest Concentration: The highest concentration extends from the second knuckle of the trigger finger, back over the web of the hand to the wrist area.

Smith & Wesson K22 Model 16



Figure 4.13 Smith & Wesson K22 Model 16 (.22 Long Rifle).

History: This firearm is a typical .22-caliber target revolver.

Description: This revolver has a 6-shot cylinder and a 6-in. barrel. It is built on the K frame. The firing pin is separate from the hammer, built into the frame, inertia type.

Objective: To show the effect of the .22 in a longer barrel.

Area of Highest Concentration: A very light concentration in front of the third knuckle of the trigger finger, extending back beyond the web and midway to the wrist.

Colt Single Action



Figure 4.14 Colt Single Action (45 caliber).

History: This revolver was designed in 1873 and is still manufactured by Colt. Several copies are currently made in the U.S. and Italy.

Description: This single action revolver has a 6-shot cylinder and a 5¹/₂-in. barrel. The firing pin is on the hammer.

Objective: To show the effect of a single-action revolver.

Area of Highest Concentration: Engulfs the entire shooting hand, blowing backward to the wrist.

Remington 870 Express

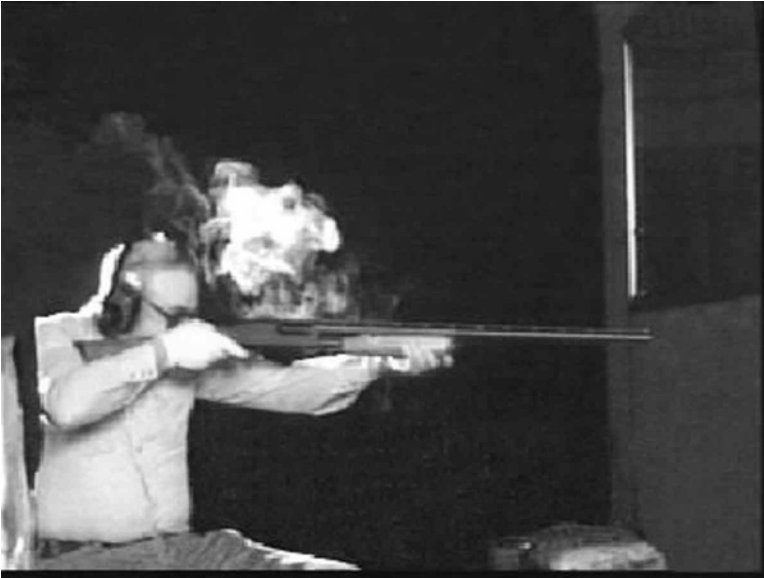


Figure 4.15 Remington 870 Express (12 gauge).

History: This very common weapon is representative of the pump shotguns manufactured in the U.S.

Description: This shotgun is plugged to 3-round capacity and has a 90° ejection port to the right.

Objective: To illustrate a pump shotgun.

Area of Highest Concentration: After a light puff toward the face on discharge, a heavy plume develops over the inner support arm, shooting hand, backward over the shoulder, face, and hair upon cartridge ejection.

Postal Meter M-1 Carbine



Figure 4.16 Postal Meter M-1 Carbine (30 carbine).

History: This firearm is essentially a reduced-size Garand action. A U.S. military weapon from World War II, it is still made commercially in the U.S.

Description: This semi-automatic rifle has a 15-shot clip (a 30-shot clip is also available) and an 18-in. barrel. The bolt opening is 180° with the ejection to the right.

Objective: To illustrate the use of a common weapon that has had some police use in the U.S.

Area of Highest Concentration: From the crook of the support arm, hand, and backward over the shooting hand, face, forehead, and hair.

Thompson 1927 A-1

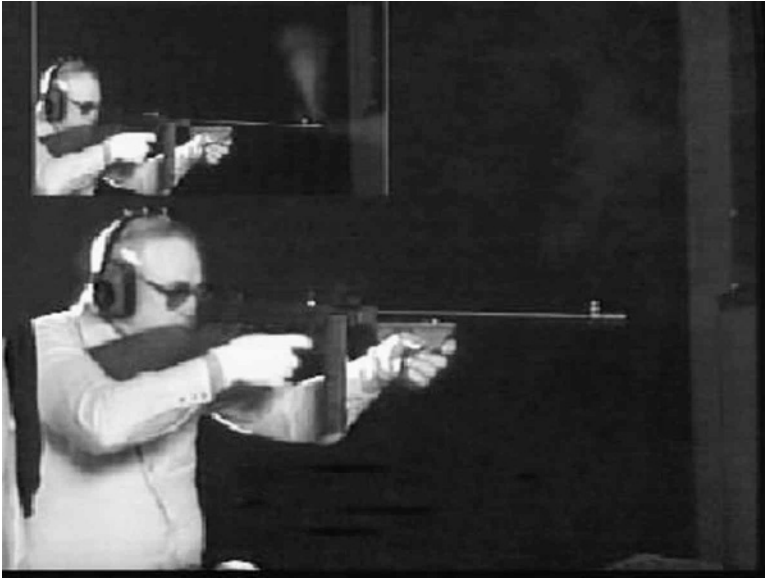


Figure 4.17 Thompson 1927 A-1 (45 ACP).

History: This is a semi-automatic version of the Thompson Sub-Machine Gun that is currently produced by Auto Ordnance Company.

Description: This rifle has a 20-round stick (a 50-round drum magazine is also available) with a 16-in. barrel. The top ejection port is open through a slot, not a full 180° opening.

Objective: To show the effect of a Thompson Sub-Machine Gun.

Area of Highest Concentration: From just behind the wrist of the support arm, backward over the support arm, to the face, forehead, and hair.

Colt AR 15



Figure 4.18 Colt AR 15 (223 caliber).

History: This model is the semi-automatic version of the current U.S. Military M-16.

Description: This semi-automatic rifle has a 30-shot clip with an 18-in. barrel. The side ejection port is 90° to the right.

Objective: To show the effect of the current M-16 style rifle using the current U.S. 223 military cartridge.

Area of Highest Concentration: The heaviest concentration is along the inside of the support arms and back to the face, forehead, and hair.

Boretta BM 59



Figure 4.19 Boretta BM 59 (308 caliber).

History: The BM 59 is an Italian-manufactured Garand copy that has been converted to a detachable magazine model. This weapon is a commercial semi-automatic version of the same model used by the Italian army. It uses the standard NATO 308 rifle cartridge.

Description: This rifle has a 20-shot clip and an 18-in. barrel. The Garand bolt has a 180° port opening with the ejection to the right.

Objective: To show an assault weapon with the NATO cartridge.

Area of Highest Concentration: Along the inside of the support arm, over the shoulder, face, forehead, and hair.

Remington Model 550 Semi-Automatic



Figure 4.20 Remington Model 550 Semi-Automatic (.22 Long Rifle).

History: This weapon is a typical .22-caliber semi-automatic rifle.

Description: This long rifle has a 15-shot tubular magazine and a 24-in. barrel length. The ejection port is 90° to the right.

Objective: To show the effect of a .22 rifle.

Area of Highest Concentration: A very light concentration over the crook of the support arm and very light wispy puffs over the shooting hand, face, forehead, and hair.

Norinco Model SKS

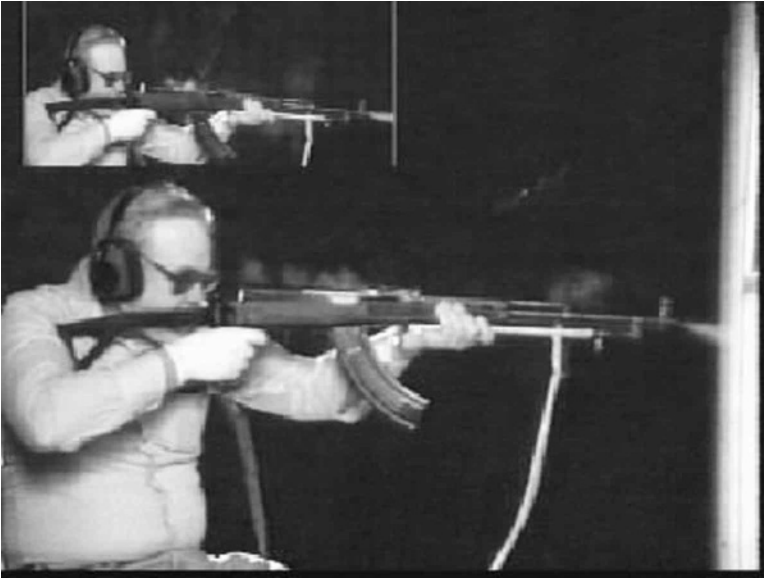


Figure 4.21 Norinco Model SKS (7.62 × 39mm caliber).

History: This weapon has a replaced stock and a 30-shot detachable clip. As manufactured, the SKS has a fixed 10-shot magazine. This is an inexpensive, reliable, widely available rifle. It is manufactured in China.

Description: This semi-automatic rifle has a fixed 10-shot magazine with a 20-in. barrel length and a 180° port.

Objective: To illustrate a high-capacity magazine weapon in the 7.62 × 39mm cartridge.

Area of Highest Concentration: The plume forms along the support arm to the face and hair in a backward blowing direction.

Winchester 94



Figure 4.22 Winchester 94 (30-30).

History: This is a very common weapon in the U.S, used by many police departments.

Description: This is a lever-action rifle with top ejection and exposed hammer. It has a 7-shot tubular magazine and a 20-in. barrel length.

Objective: To show the effect of a top-ejection, lever-action rifle.

Area of Highest Concentration: A slight wisp behind the chamber and near the web of the shooting hand and a light distribution over the support arm, spreading to the face and hair, upon cartridge ejection.

Marlin 444S



Figure 4.23 Marlin 444S (444 caliber).

History: This lever-action rifle is typical of the Marlin line of lever-action rifles.

Description: This rifle has a 4-shot tubular magazine and a 22-in. barrel length. It is a lever-action rifle with a side ejection port. The hammer is exposed and the ejection port is on the right side.

Objective: To show the effect of a side-ejection, lever-action rifle.

Area of Highest Concentration: A heavy concentration over the support arm, spreading to the face, forehead, and hair upon cartridge ejection.

Savage 99



Figure 4.24 Savage 99 (300 Savage).

History: This rifle was the first commercial hammerless lever action rifle. It has been in production for 100 years.

Description: This lever-action rifle has a rotary 5-round magazine with a 24-in. barrel length. It has a 120° ejection port on the top and right side. The hammer is concealed.

Objective: To show the effect of a hammerless, lever-action rifle.

Area of Highest Concentration: From the crook of the support arm and backward over the hand, face, forehead, and hair upon cartridge ejection.

Winchester 70

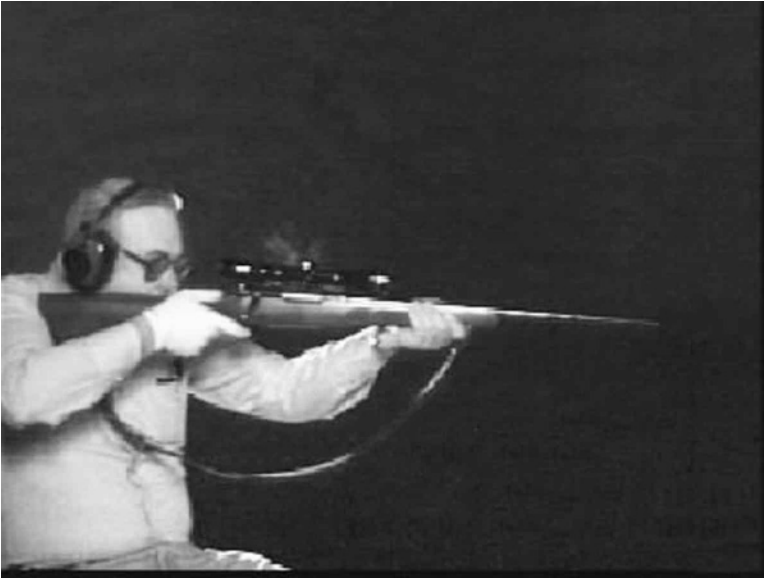


Figure 4.25 Winchester 70 (308 caliber).

History: This firearm is typical of the bolt-action rifle.

Description: This bolt-action rifle has a 5-shot magazine and a 22-in. barrel length. The ejection port opens to 90° and ejects from the right.

Objective: To show the effect from a typical bolt-action rifle.

Area of Highest Concentration: Centered over the crook of the support arm and extending slightly backward over the face, forehead, and hair, upon cartridge ejection.

Remington 760 Pump Rifle

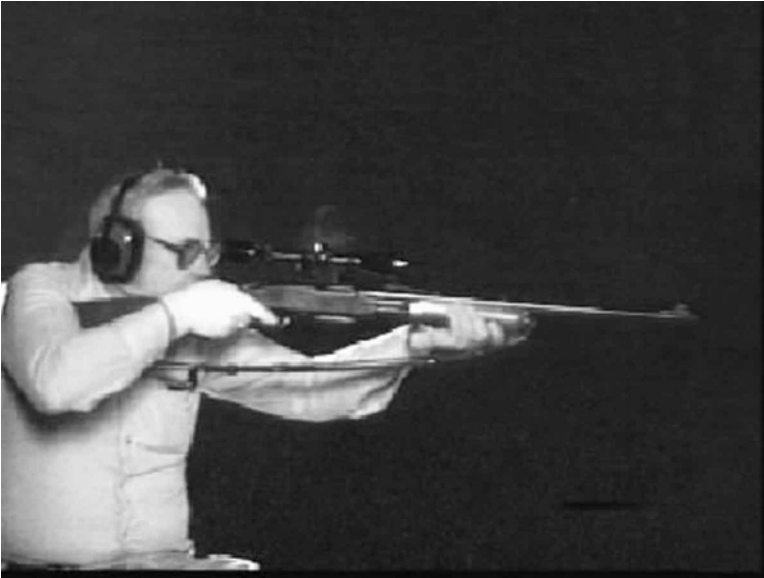


Figure 4.26 Remington 760 Pump Rifle (35 Remington).

History: This firearm is a typical pump rifle.

Description: This firearm has a 4-shot detachable clip and a 22-in. barrel length. The ejection port is 90° to the right.

Objective: To show the effect of a pump rifle.

Area of Highest Concentration: A light distribution directly over the support arm during the ejection of the cartridge.

LaFever Nitro Special



Figure 4.27 LaFever Nitro Special (16 gauge).

History: This is a typical double-barrel shotgun.

Description: This shotgun is 2-shot with a 28-in. barrel length.

Objective: To show the effect of a double-barrel shotgun.

Area of Highest Concentration: No plume is visible on firing; however, when cartridges are removed immediately after firing, a large plume spreads over the hands, clothing, face, and hair.

Remington 1100



Figure 4.28 Remington 1100 (12 gauge).

History: This shotgun is a typical U.S. manufactured semi-automatic shotgun.

Description: It is plugged to 3-round capacity and has a 30-in. barrel. The ejection port is 90° to the right.

Objective: To illustrate the effect of an auto-loading shotgun.

Area of Highest Concentration: A light concentration over the support hand and a heavier distribution over the crook of the support arm, spreading to the face and hair during cartridge ejection.

Summary of Results

The significance of the plume study lies in the various types of firearms available and the differences in the plumes that are created. When evaluating the likelihood of gunshot deposition on surrounding surfaces (forearm, face, etc.), information gained from test firing various weapons and characterizing the resulting plume becomes important.

Smaller caliber semi-automatic handguns with high or forward cartridge ejection have plume concentrations toward the front of the fingers in some instances, which are heavier than web and wrist areas. In most cases, the plume tends to follow the direction of cartridge ejection. Larger caliber revolvers have a widespread plume as opposed to the more compact plume of larger caliber semi-automatic weapons with ejection ports.

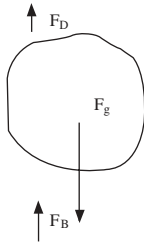
The most consistent area of plume concentration for rifles and shotguns is the crook of the support arm; however, blow-back or drift of the original plume formation is toward the chest, shoulder, face, and hair, with heavy concentrations for some weapons and light for others. Again, cartridge ejection is a factor in many of these weapons. Even when influenced by the direction of cartridge ejection, the plume expands quickly in all directions and can also be influenced by turbulent air in the vicinity of discharge.

Particle Fallout

How long a GSR particle will remain in the environment of a discharging firearm is a common question. Is there a certain amount of time particles of GSR will stay airborne and potentially contaminate individuals who enter the vicinity of a weapon discharge after the fact? In an attempt to shed light on this issue, an approximation of how long 1-micron (μm) GSR-like particles remain airborne (in the plume) was undertaken.

A first-order approximation of particle settling velocities can be obtained by the application of Stokes law. This relationship assumes static conditions and ignores turbulent factors. The purpose of this exercise is to identify the differences between the fallout time of a sphere compared to a disk. Therefore, a form of Stokes law containing a term for the coefficient of aerodynamic drag was determined. A force diagram can be found in the following examples.¹ The buoyancy and drag forces are illustrated opposing the gravitational force.

For Sphere:



$$F_D \equiv \text{Drag Force} = \frac{1}{2} C_D A_p \rho_L V^2$$

$$F_g \equiv \text{Gravitational Force} = \rho_p g V_p$$

$$F_B \equiv \text{Buoyancy Force} = \rho_L g V_p$$

$$A_p \equiv \pi r^2$$

Particle acceleration is driven by a differential between F_g and F_B .

$$F_g - F_B = (\rho_p - \rho_L) g V_p \quad V_p = \frac{4}{3} \pi r^3$$

When the drag force equals the force driving acceleration the terminal velocity of the particles is reached:

$$\therefore F_g - F_B = F_D$$

Substitution gives the following relationship:

$$\frac{4}{3} (\rho_p - \rho_L) g \pi r^3 = C_D A_p \rho_L \frac{V_s^2}{2}$$

ρ_p = particle density
 ρ_L = medium density
 d = particle diameter
 g = gravitational acceleration
 C_d = coefficient of drag (Reference 2)

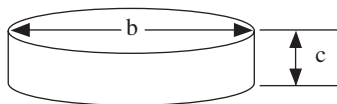
Now solving for V_s yields the terminal settling velocity for the particle in a given medium of determined density.

$$V_s = \left[\frac{4}{3} \left(\frac{(\rho_p - \rho_L) g d}{C_D \rho} \right) \right]^{1/2}$$

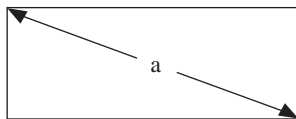
In this form, Stokes law was used to calculate the terminal settling velocities of spherical particles. For the disk-shaped particles, a substitution function was applied in place of the diameter (3). The following shows the relationship of equivalent diameter to the geometry specific measurements of a given particle. In this example a disk geometry was assumed:

For Disk:

$$d = \frac{c}{\sqrt{ab}}$$



- @ a ≡ longest dimension
- b ≡ intermediate dimension
- c ≡ shortest dimension
- d ≡ equivalent dimension



The value resulting from the equivalent diameter calculation was used for the disk terminal velocity calculations.

After a terminal velocity was calculated, the time and distance required for the particle to reach terminal velocity were calculated to estimate the total time that the particle remains in suspension. The equations of motion 4 from particle dynamics were used to calculate the time as follows:

$$V = V_0 + at \quad \text{where } a \rightarrow c_D g$$

$$(a) \quad t_0 = \frac{V - V_0}{c_D g} \Rightarrow \textit{Time to Terminal Velocity}$$

$$(b) \quad Y = \frac{1}{2}(V_0 + V)t \Rightarrow \textit{Distance Traveled to Terminal Velocity}$$

The value collected from Equation (a) was added to the fallout time (found by dividing the fallout distance by the terminal velocity). The summation of these values determined the estimated fallout time for the particles of given geometry. Following are calculations showing the variations obtained for particles of similar density, but different shape as well as same shaped particles with different densities. An initial height of 3 meters (m) was assumed:

For 3 m height:

$$\frac{3 \text{ m} - Y}{V_s} + t_0$$

$$Pb \text{ sphere} \rightarrow \frac{3 \text{ m} - Y}{V_s} + t_0 = \frac{3 \text{ m} - 0.28 \text{ m}}{0.515 \text{ m/s}} + 0.11 \text{ s} = 5.9 \text{ s}$$

$$Pb \text{ disk} \rightarrow \frac{3 \text{ m} - 0.0024 \text{ m}}{0.231 \text{ m/s}} + 0.021 \text{ s} = 13.0 \text{ s}$$

$$Ba \text{ sphere} \rightarrow \frac{3 \text{ m} - 0.0089 \text{ m}}{0.286 \text{ m/s}} + 0.062 \text{ s} = 10.5 \text{ s}$$

$$Ba \text{ disk} \rightarrow \frac{3 \text{ m} - 0.001 \text{ m}}{0.129 \text{ m/s}} + 0.012 \text{ s} = 23.3 \text{ s}$$

EXAMPLE: Lead sphere 1 μm in diameter falling from a height of 3 m, at room temperature with no turbulence.

- 1) The terminal velocity is found from:

$$V_s = \sqrt{\frac{4g(\rho_p - \rho_L)d}{3C_0\rho_L}}$$

where: g = acceleration of gravity 9.8 m/s²

ρ_p = particle density

ρ_L = medium density

d = particle diameter, μm

C_0 = coefficient of drag

$$V_s = \left[\frac{4(9.8 \text{ m/s}^2)(11,000 \text{ kg/m}^3 - 1.185 \text{ kg/m}^3)(1 \times 10^{-6} \text{ m})}{3(0.47)(1.185 \text{ kg/m}^3)} \right]^{1/2}$$

$$V_s = \left[\frac{0.443 \text{ kg/ms}^2}{1.67 \text{ kg/m}^3} \right]^{1/2} = \left[\frac{0.443 \text{ m}^2}{1.67 \text{ s}^2} \right]^{1/2}$$

$$V_s = 0.515 \text{ m/s}$$

- 2) Time for the lead sphere to reach terminal velocity.

$$t_0 = \frac{V_s - V_0}{C_0 g} = \frac{0.515 \text{ m/s} - 0}{(0.47)(9.8 \text{ m/s}^2)}$$

$$t_0 = 0.11 \text{ s}$$

- 3) The distance traveled to reach terminal velocity is calculated as follows:

$$Y = \frac{1}{2} (V_0 + V) t$$

$$Y = \frac{1}{2} (0 + 0.515 \text{ m/s}) 0.11 \text{ s}$$

$$Y = 0.28 \text{ m} = 28 \text{ mm} = d_0$$

- 4) The total time that the particle remains in suspension:

$$T_{\text{total}} = \frac{d_T - d_0}{V_s} + t_0$$

$$T_{\text{total}} = \frac{3 \text{ m} - 0.28 \text{ m}}{0.515 \text{ m/s}} + 0.11 \text{ s}$$

$$T_{\text{total}} = 5.88 \text{ s}$$

Conclusions

This exercise shows that particle fallout times can vary widely, based upon the physical characteristics of the subject particle. Given the same size and shape, particles of higher density will settle out faster than those of less dense composition. The particles generally associated with the classification of GSR are among the densest materials known (e.g., lead). These materials would be expected to settle out of the air relatively quickly after the firing incident.

Note that extenuating factors exist, on site, that would certainly change the settling times estimated in the previous calculations. Factors such as turbulence, temperature, humidity, and pressure would affect the length of time that a particle would remain airborne. Generally, an increase in these factors will probably increase the entrainment time.

References

1. Davis, M. and Cornwell, D., *Introduction to Environmental Engineering*, 2nd ed., McGraw-Hill, New York, 1991, 199.
2. Eshback, O., *Handbook of Engineering Fundamentals*, 2nd ed., John Wiley & Sons, New York, 1953, 7–103.
3. Syvitski, J. P. M., *Principles, Methods, and Applications of Particle Size Analysis*, Cambridge University Press, Cambridge, 1991, 12.
4. Holiday, D. and Resnick, R., *Physics: Parts I and II Combined*, John Wiley & Sons, New York, 1978.

To understand how gunshot residue (GSR) is deposited, one must understand the various types of firearms and how they affect the way GSR is created and deposited in the vicinity of the discharge. The compositions of the ammunition components — cartridge casing, bullet, and primer — help us understand why various particles have characteristic elemental composition and morphology. This chapter describes the various firearms used today and the ammunition components that play major roles in the creation of the particles known as GSR.

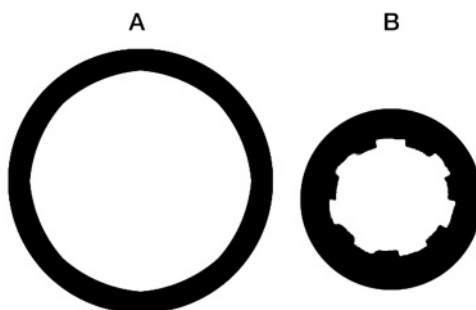


Figure 5.1 Schematic view of the internal bore characteristics of (A) a smooth-bore barrel and (B) a rifled barrel.

Firearms

Two main types of firearms will be discussed: rifled and smooth-bore (Figure 5.1). In rifled firearms, the bore contains a number of grooves parallel to one another and cut with a spiral twist from the breech to the muzzle. The lands are the high places between the grooves. The caliber is determined by the internal diameter of the barrel. In contrast, smooth-bore firearms have a perfectly smooth bore from one end to the other. The gauge of smooth-bore barrels is determined by the number of spherical balls of pure lead that are needed to make up 1 lb. Each ball fits separately and exactly inside the bore.



Figure 5.2 Winchester Model 70 308 bolt action rifle.



Figure 5.3 M1 carbine 30 carbine semi-automatic rifle.

Rifles

The typical description of a rifle is “a firearm with a long-rifled barrel and a shoulder stock.” Rifles are categorized as manual or semi-automatic, depending on the reloading mechanism. In a manual rifle (Figure 5.2), the ejection of the spent case and loading of a new cartridge is accomplished by hand operation of a bolt, lever, or slide. A semi-automatic rifle (Figure 5.3) uses the gas or recoil of the gun to eject the spent casing and load a new cartridge into the chamber each time the trigger is pulled.

Shotguns

The shotgun, like the rifle, is a shoulder-stocked gun with a long barrel (except in the case of a sawed-off barrel); however, the shotgun barrel is usually a larger diameter and not rifled (Figure 5.4). Another major difference is the type of ammunition used. Rifle ammunition (Figure 5.5) consists of a casing, primer, and bullet, whereas shotgun ammunition is usually composed of lead pellets rather than a single bullet. Shotshells (Figure 5.6) consist of shot pellets (buckshot or slug load), shotshell casing (cardboard or plastic tube), primer, powder charge, and wads (cardboard, plastic, or felt).



Figure 5.4 REM model 58 12-gauge semi-automatic shotgun.

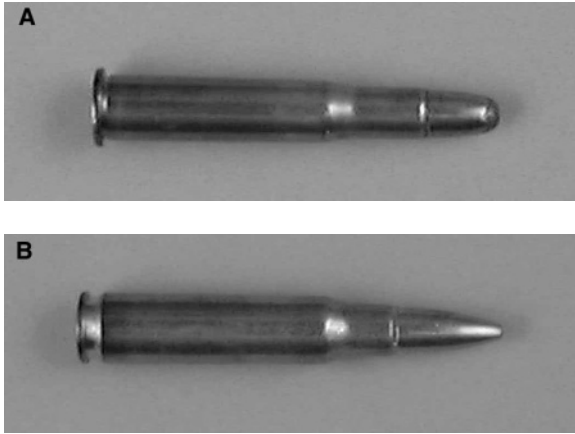


Figure 5.5 Examples of rifle ammunition: (A) 30-300 ammunition and (B) 308 ammunition.

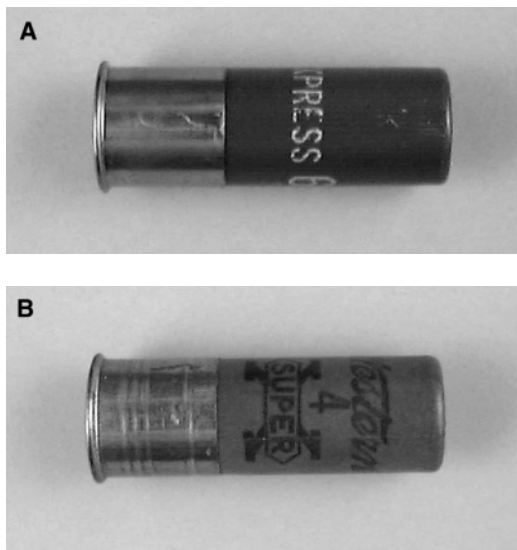


Figure 5.6 Examples of shotgun ammunition: (A) 12-gauge plastic and (B) 12-gauge paper.

Pistols

The pistol is a firearm with a short-rifled barrel that is fired with one hand. There are three general types of pistols: single-shot, revolver, and semi-automatic.

The single-shot pistol allows the shooter to fire one shot at a time. It must be manually reloaded each time.

The revolver (Figure 5.7) has a revolving cylinder, which contains many firing chambers. After a shot is fired, the chamber revolves, placing another chamber in line with the firing pin, thus allowing the shooter to fire another shot without manually reloading. This process can be continued until all of the bullets in the chambers are fired. Some revolver models have up to ten firing chambers. In a single-action revolver, the firing chamber revolves to align with the firing pin when the hammer is manually cocked back with the thumb. In a double-action revolver, as the trigger is pulled, the hammer is automatically raised while the chamber lines up and the force of the continued trigger pull fires the pistol.

The semi-automatic firearm (Figure 5.8) is very different from the single-shot pistol and the revolver. It contains a magazine that automatically loads the ammunition into the chamber following the discharge. The mechanism of the semi-automatic pistol also discards the expired cartridge casing by means of an extractor. As the breechblock moves back after the firing of the ammunition, the ejector expels the spent casing. The significance of the semi-automatic pistol to the examiner is that the spent casings that are ejected can be recovered from the scene for subsequent comparison with known spent casings from the same firearm. The manual action of the single-shot pistol and revolver leaves no such evidence behind.

Cartridges

The Cartridge Case

The entire live round of ammunition containing the primer, gunpowder, casing, and the projectile is called the cartridge. In shotgun ammunition, the cartridge also contains a wad. The cartridge casing is the metal casing that holds together all the components of the cartridge. It is most commonly composed of brass, which is 70% copper (Cu) and 30% zinc (Zn), and referred to as “cartridge brass.”¹ Brass is used rather than other metals because of its performance, cost, and ease of manufacturing. Other types of metals have been used to make cartridge casings, including aluminum (Al), Zn, Cu, steel, and some types of plastic. During World War II, steel casings were manufactured for .45 caliber pistol ammunition in the U.S. and for 8mm ammunition manufactured in Germany. Today, some ammunition is still manufactured with steel cartridge casings.



Figure 5.7 Examples of typical revolvers: (A) S&W model 36 and (B) S&W model 629.

The reason brass is the metal of choice in the manufacture of most cartridge casings is easy to understand when brass casings are compared to steel casings. Although steel is much cheaper (about a quarter of the cost), steel rusts and also abrades the chamber. Another reason brass is used more often is the cost of the manufacturing process. Steel is more difficult to work with and requires special lubrication. This results in increased wear on manufacturing equipment, which offsets the cost of the raw material. Another disadvantage to using steel relates to the formation of the casing, because steel is more difficult to work with than other metals. Other ratios of Cu and brass have been used in the manufacture of cartridge casings, with variable results. Although Cu alone is a good material for forming the casing, it lacks



Figure 5.8 Examples of semi-automatic pistols: (A) Colt 1911 A1 and (B) Ruger MK11.

the strength to withstand the high pressure of smokeless powder loadings. Pure Zn has been shown to be of very little value because it is too soft. However, when Cu and Zn are combined in the correct proportions, a workable casing that meets the requirements of a high-pressure casing is formed.

The two main types of cartridges are rimfire ammunition and centerfire ammunition (Figure 5.9).

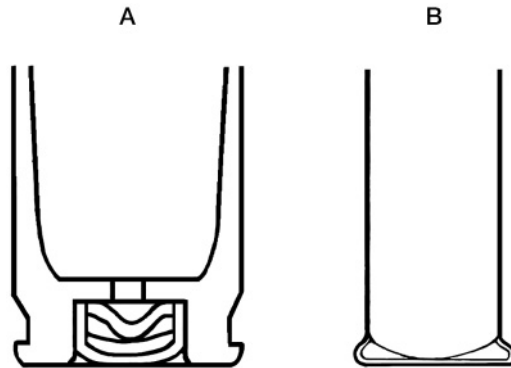


Figure 5.9 Examples of (A) centerfire cartridge and (B) rimfire cartridge.

Rimfire Cartridge

Rimfire ammunition is the most commonly used type of ammunition. Several billion rounds of .22 rimfire ammunition are manufactured annually; it is the ammunition of choice for most Americans. The term “rimfire” is associated with .22-caliber ammunition. The .22 rimfire ammunition case is mostly composed of a brass case (steel .22 rimfire cartridges are still manufactured in Russia). The four basic types of rimfire cartridges are .22 Short, .22 Long, .22 Long Rifle (Figure 5.10), and .22 Winchester Magnum. The priming compound is contained in the rim area of the casing around the cartridge. When the firing pin strikes the face, the rim collapses, causing the back and forward edges to crush the priming compound. This reaction ignites the priming compound and initiates a flame that ignites the propellant.

Centerfire Cartridge

The priming compound of the centerfire cartridge is located in self-contained primer cups in the center of the head end of the cartridge (Figure 5.11). Centerfire cartridges are commonly used in .38 Special caliber weapons.

Cartridge Casing

The cartridge casing is the container that holds all of the cartridge components together. There are five subtypes of cartridge casing: rimmed, semi-rimmed, rimless, belted, and rebated. Rimmed cases have a full rim at the base of the cartridge case, larger in diameter than the diameter of the rest of the body of the cartridge. This flange enables the cartridge to be extracted from the weapon. Rimmed cases are commonly used in revolver cartridges. They are designated with the letter “R” after the case length measurement.

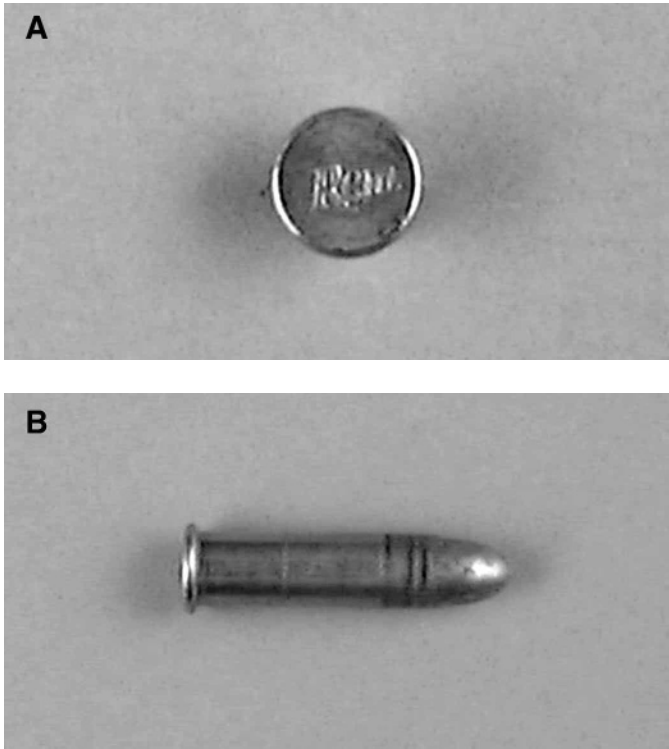


Figure 5.10 An example of .22 rimfire ammunition: (A) .22 headstamp and (B) longitudinal view.

Semi-rimmed (“SR”) cases exhibit a slightly larger rim than the cartridge case. The .25 auto cartridge typifies this type of case. Rimless cases, commonly found in the .45 auto cartridge, have no rim around the base; they have a groove around the case body just in front of the base to produce a flange for extraction purposes. There is no letter designation for rimless cases. Belted (“B”) cases have an area that is built up or reinforced at the base of the cartridge, providing additional strength in high pressure cartridges for high-powered rifles and some military ammunition. Rebated cases (“RB”), which have an extractor flange of a diameter less than that of the cartridge case, are used in a few rifle cartridges. The shape of the cartridge case may be straight, tapered, necked, or tapered necked.

Bullet

The bullet is the nonspherical projectile that is used in a rifled barrel. Bullet design is based on several factors, including the powder charge, type of



Figure 5.11 Example of .45 ACP centerfire ammunition.



Figure 5.12 Example of full metal jacket bullet.

firearm for which is it designed, and ballistics. Bullets come in a variety of compositions and constructions. A few common types will be discussed briefly, but this is by no means a comprehensive description of the various types of bullets that are manufactured and used today.

Jacketed and unjacketed bullets are the most common types of bullets used in small arms ammunitions. Jacketed bullets are usually made with an inner core of Pb covered with a harder material such as Cu/Zn, Cu/Ni, or

steel with a Cu wash (Figure 5.12). Other types of small ammunition bullets are nylon-coated Pb bullets (“Nyclad”) or Pb bullets with a wash of Cu (“Luballoy”). Frangible bullets, usually composed of Cu and synthetic polymer, are also commercially available. They are widely used for “lead-free” ammunition and on firing ranges.

Powder Charges

There are several types of powder charge or “propellant.” Black powder, composed of charcoal (15%), sulfur (10%), and potassium nitrate (75%), is seldom used today. The most commonly used propellants today are single- and double-based smokeless powders. Single-based powders are composed of nitrocellulose. Double-based powders are a combination of nitrocellulose and nitroglycerin.

Primer

Three major components in the primer compound play major roles in the formation and identification of GSR:

1. The initiator: Lead styphnate, which is set off with a sharp blow, as when the firing pin strikes the primer cap.
2. The oxidizer: Barium nitrate, which gives up its oxygen readily. The oxygen is needed to burn the fuel.
3. The fuel: Antimony sulfide, which burns at a very high rate. This initial burn ignites the gunpowder, which propels the bullet.

Other common components of the primer compound are vital in the role of the ammunition. The sensitizer is added to the styphnate to make the mix irritable, due to the sluggish nature of the styphnate. The most common type of sensitizer is called “tetracene” or guanyl nitrosoaminoguanyl tetracene. The frictionator, needed for rimfire primers, is commonly composed of ground glass. The sharp corners of the frictionator provide a force to the sensitizer and initiator that compresses and ruptures explosive crystals. Binders are used to hold the primer mix in place. Various gums, such as gum arabic and gum tragacanth, starches, polyvinyl alcohol, and other materials are used as binders. Coloring materials are sometimes used in the primer compounds. Explosives such as PETN, TNT, and DDNP, may be used to provide heat and energy to the reaction.

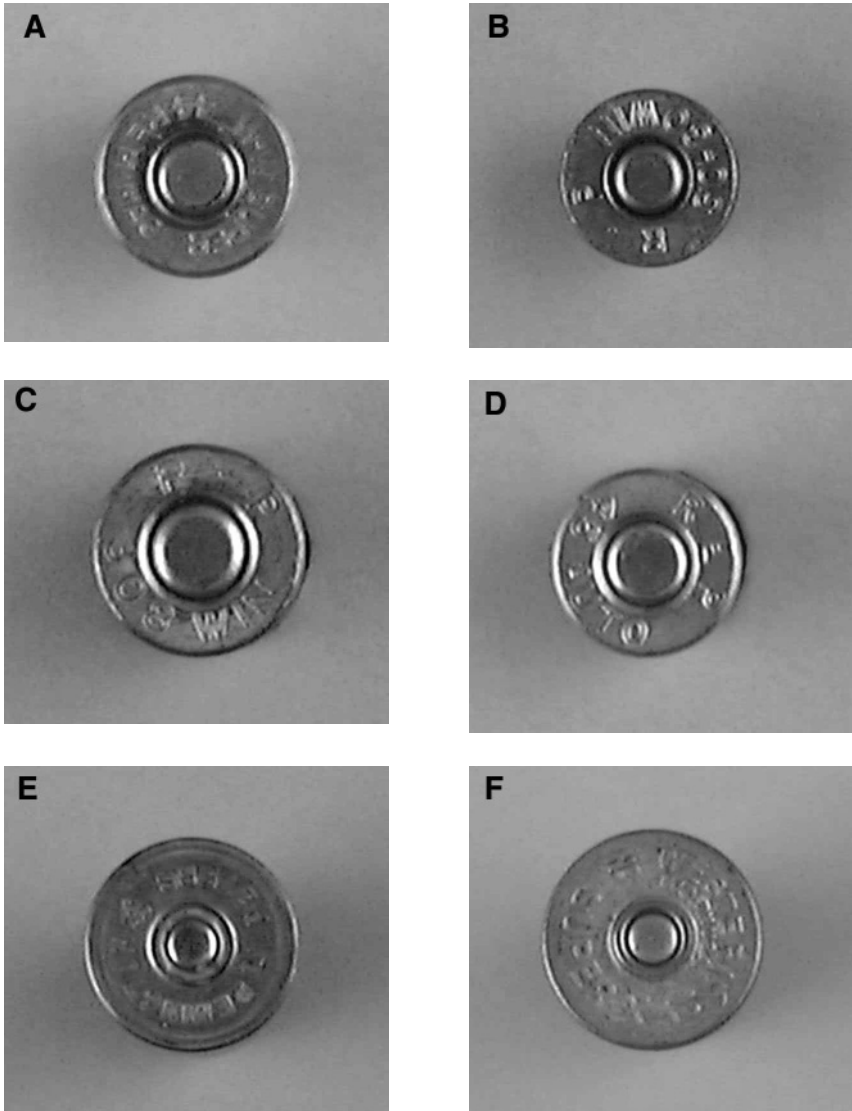


Figure 5.13 Examples of various head stamps.

Head Stamps

Most manufacturers stamp markings into the head of the cartridge to designate the manufacturer's name and caliber type. [Figure 5.13](#) shows six examples of head stamps for various types of ammunition. Military markings may include letter codes for the manufacturer and single or double digits for year of manufacture.

.22 Caliber Ammunition Composition

Manufacturers of primers are not required to disclose their formulations or any changes they may make in their formulations. This information is considered proprietary and not made available to researchers and examiners. The only way for examiners to keep abreast of primer formulation changes is to conduct test firings of known ammunition and report their findings. The 1989 change in some formulations of .22 Remington rimfire ammunition is a case that underscores the importance of examiners conducting tests and reporting their results. Some analysts who still adhered to the findings of the Aerospace Report of 1977 did not routinely analyze .22 Remington rimfire ammunition because it was common knowledge in the field that this formulation did not contain Sb or Ba. However, in 1989, Remington added Sb and Ba to its .22 rimfire primer, which meant GSR examination methods could now detect Sb and Ba in this ammunition. The only way analysts could learn about this change was through their own routine analyses of .22 Remington rimfire ammunition. It is vital that examiners conduct routine analysis on all primers and that they keep abreast of the findings reported by others.

Ammunition Composition Studies

In 1998, Wrobel et al.² published results of their attempt to identify the ammunition from GSR and cartridge materials using .22 rimfire ammunition. A prototype database of chemical and physical characteristics was created to identify 70 types of .22 caliber ammunition.

At RJ Lee Group laboratories, we have attempted since 1993 to characterize various types of GSR from various types of ammunition and firearms to determine if any significant information could be compiled.

GSR Studies: U.S. Manufacture Pistol Primers

A cartridge has four components: the case, the bullet, the powder, and the primer. Each component plays a role in the elemental composition of GSR particulate.

The case is generally manufactured from brass (Cu, Zn) and may be Ni plated. Brass is not usually found in GSR.

Pistol bullets may be Pb based with small amounts of Sb or tin; they are swaged or cast. Some of these Pb bullets are plated with Cu or brass. Pistol bullets may also be jacketed with Cu or brass. Pb-only particles in GSR or Pb plus Cu or brass particles may indicate bullet particles.

Modern smokeless powders contain light elements (e.g., carbon, oxygen, and nitrogen), which are not detected by scanning electron microscopy (SEM).

The primer consists of a small brass or Ni-plated brass cup with a thin layer of priming compound topped with a foil seal and an anvil made of brass. U.S. pistol primers are manufactured in two diameters: small pistol (0.175 in.) and large pistol (0.210 in.). For the past 25 years or more, the priming composition has contained Pb generally as lead styphnate, and usually Ba and Sb; Ba is usually added as barium nitrate and Sb is added as antimony sulfide. Aluminum (Al), calcium (Ca), and silicon (Si) may also be present.

Compositions considered unique to GSR are Pb, Sb, and Ba. These compositions may contain additional elements. The occurrence of Pb/Sb, Pb/Ba, Sb/Ba, or Ba/Ca/Si/Trace S, although not unique to GSR, are characteristic of it.

Since the primer is the component that is the major contributor to GSR, it was decided to isolate the primer effect as much as possible and to examine the various primers manufactured in the U.S. This study was conducted to aid law enforcement personnel by providing comparative data on known reference materials. Four U.S. primer manufacturers currently produce six different large pistol primers and seven different small pistol primers. The primer types are as follows:

<u>Large Pistol Primers</u>	<u>Small Pistol Primers</u>
CCI 300	CCI 500
CCI 350M	CI 550M
Federal 150	Federal 100
Federal 155 Mag	Remington 1.5
Remington 2.5	Remington 5.5

To isolate the primer effect, these primers were hand loaded into new cases with jacketed hollow-point bullets. The same powder was used for all loads regardless of caliber. The new case eliminated any contamination from primer residue in cases. Jacketed hollow-point bullets were selected since the base, side wall, and part of the ogive are covered with a jacket of Cu or brass, and will, therefore, minimize the Pb bullet particles in the GSR. Using a single powder should produce similar powder flake remnants in all the tests.

The powder selected for the test loads was Hercules Powder Company's Unique, a double-base powder containing both nitrocellulose and nitroglycerin. This powder was selected because it can produce loads with velocities similar to factory loads without developing excessive pressures. The following loads used for the tests were selected after reviewing data in a several hand-loading manuals.³⁻⁵

Cartridge	Case	Powder weight	Bullet	Estimated muzzle velocity (fps [*])
9mm Luger	Winchester	5.5 grains	115 grain JHP Win.**	1100
357 Magnum	Winchester	8.0 grains	158 grain JHP Win.	1200
45 ACP	Winchester	6.5 grains	230 grain JHP Win.	820

* Feet per second.

** Jacketed, hollow-point Winchester.

Four handguns were used for these tests: an auto-loader and a revolver for each primer size. A Smith & Wesson Model 39 auto-loader with a 5-in. barrel and a Colt Trooper 357 Magnum revolver with a 4-in. barrel were used with the small primers. A Colt 1911A-1 45 ACP autoloader with a 5-in. barrel and a Smith & Wesson Model 1917 45 ACP revolver were used with the large primers. The handguns were fired with jacketed hollow-points and cleaned thoroughly before the tests began. This was done to remove any residue from Pb bullets and the effect of primers from previous firings. The handguns were also cleaned after each test firing to eliminate the effect of GSR from a previous firing.

One person was used for each test firing, with the stipulation that the person had not fired a gun in the past 3 months. Each test consisted of firing two rounds from each hand. Adhesive lifts utilizing adhesive samplers were taken from each hand. The right hand was sampled immediately after firing, and the left hand was sampled 24 hours later. In addition, the fired cases were de-primed, and the primer residue from the cases was examined and compared with the adhesive lifts from the hand.

Table 5.1 lists SEM images coupled with the corresponding energy dispersive spectroscopy (EDS) spectra of the particles characterized in this study. These particles are given to orient newer examiners to the sizes, shapes, morphologies, and elemental compositions of various types of gunshot primer particles produced from test firing of various ammunitions and firearms.

General Observations from Test Firings

These GSR particles resulted only from the ammunition and firearms used in this study. Much more research is needed in the area of primer particle characterization before any concrete conclusions can be reached. One major determination was made based on this study. The type of ammunition and firearm could not be determined with confidence, based solely on the morphology and composition of the resulting primer residue particulate.

Table 5.1 Gunshot Primer Particles Produced from Test Firing of Various Ammunitions and Firearms

Figure	Firearm	Cartridge	Primer
5.14–5.15	Colt MOD 1911A1	45 ACP	CCI
5.16–5.17	Colt Trooper	357 MAG	CCI
5.18–5.19	Colt Trooper	357 MAG	CCI
5.20	Colt Trooper	357 MAG	FED
5.21	S&W MOD 1917	45 ACP	CCI
5.22	Colt Trooper	357 MAG	REM
5.23	S&W MOD 1917	45 ACP	CCI
5.24–5.25	Colt Trooper	357 MAG	REM
5.26–5.27	Colt MOD 1911A1	45 ACP	FED
5.28–5.29	Colt MOD 1911A1	45 ACP	FED
5.30	Colt Trooper	357 MAG	WIN
5.31	S&W MOD 39	9mm Luger	CCI
5.32	S&W MOD 39	9mm Luger	CCI
5.33	S&W MOD 39	9mm Luger	CCI
5.34–5.35	S&W MOD 39	9mm Luger	CCI
5.36–5.37	S&W MOD 39	9mm Luger	FED
5.38–5.39	S&W MOD 39	9mm Luger	FED
5.40–5.41	Colt MOD 1911A1	45 ACP	FED
5.42–5.43	Colt MOD 1911A1	45 ACP	FED
5.44	Colt MOD 1911A1	45 ACP	FED
5.45	S&W MOD 39	9mm Luger	REM
5.46	S&W MOD 39	9mm Luger	REM
5.47	Colt MOD 1911A1	45 ACP	REM
5.48	Colt MOD 1911A1	45 ACP	REM
5.49	Colt MOD 1911A1	45 ACP	WIN
5.50	Colt MOD 1911A1	45 ACP	WIN
5.51	S&W MOD 39	9mm Luger	WIN
5.52–5.53	S&W MOD 39	9mm Luger	WIN
5.54	S&W MOD 39	9mm Luger	WIN
5.55	S&W MOD 1917	45 ACP	WIN
5.56	S&W MOD 1917	45 ACP	WIN
5.57–5.58	Colt Trooper	357 MAG	CCI
5.59	Colt Trooper	357 MAG	FED
5.60	Colt MOD 1911A1	45 ACP	CCI
5.61	Colt Trooper	357 MAG	REM
5.62	S&W MOD 39	9mm Luger	CCI
5.63	S&W MOD 39	9mm Luger	CCI
5.64	S&W MOD 1917	45 ACP	FED
5.65	S&W MOD 1917	45 ACP	REM
5.66	S&W MOD 39	9mm Luger	WIN

Note: [Figures 5.14](#) through [5.56](#) depict unique GSR (Pb-Sb-Ba); [Figures 5.57](#) through [5.66](#) depict characteristic GSR particles.

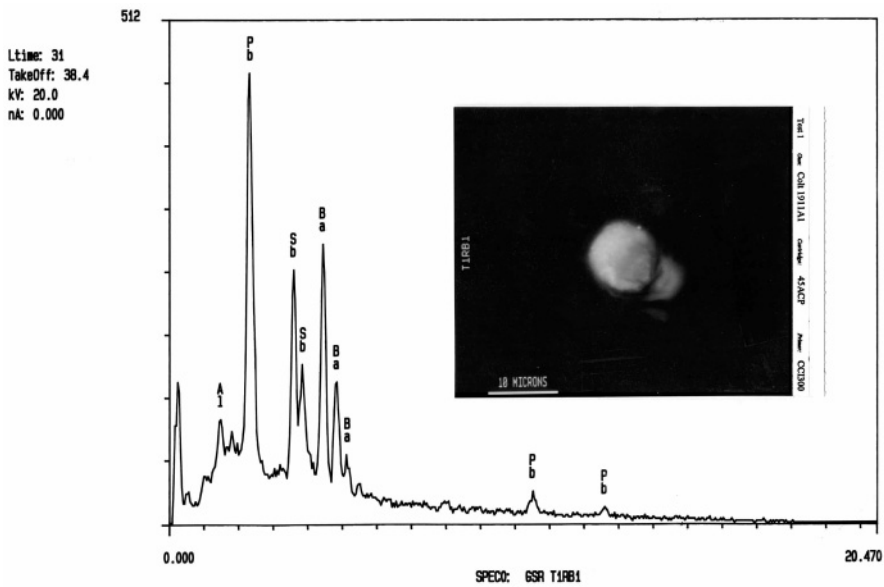


Figure 5.14 Colt MOD 1911A1 45 ACP CCI.

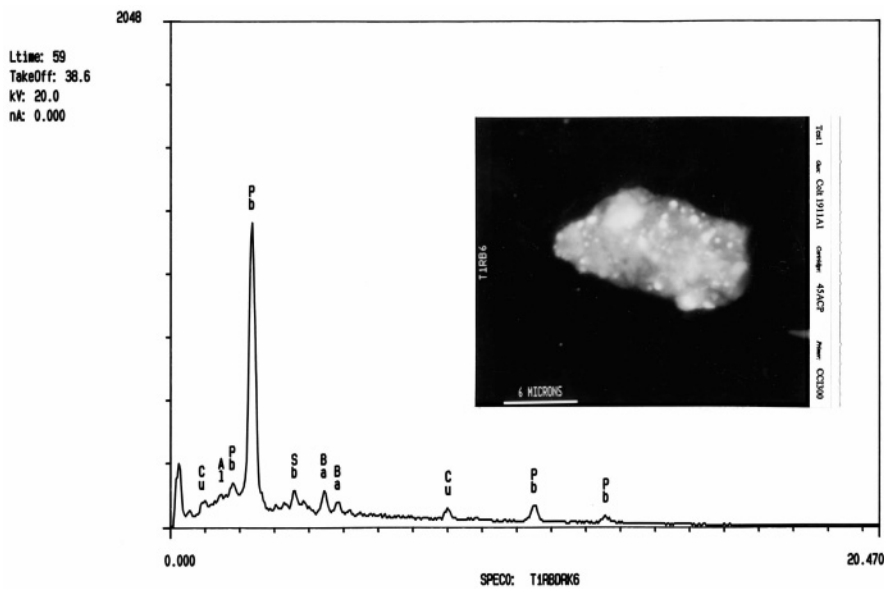


Figure 5.15 Colt MOD 1911A1 45 ACP CCI.

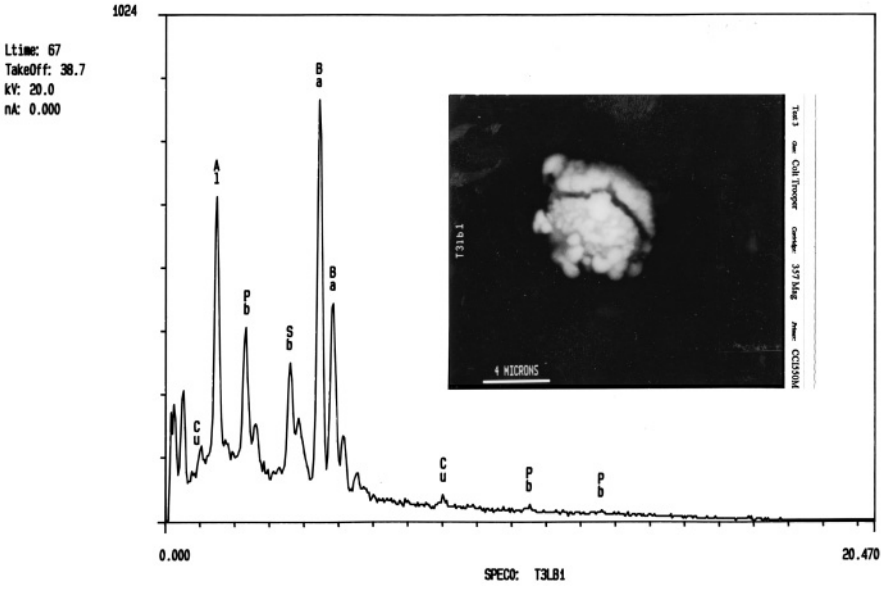


Figure 5.16 Colt Trooper 357 MAG CCI.

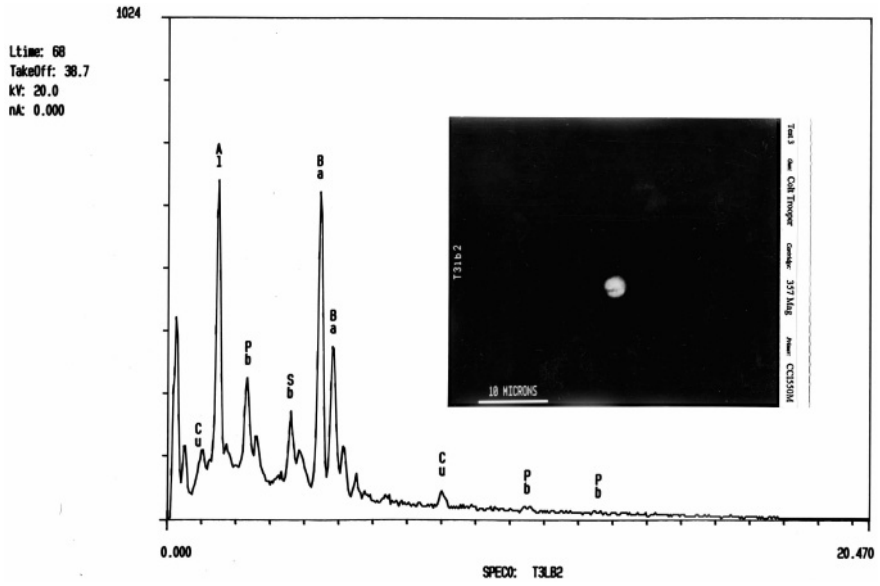


Figure 5.17 Colt Trooper 357 MAG CCI.

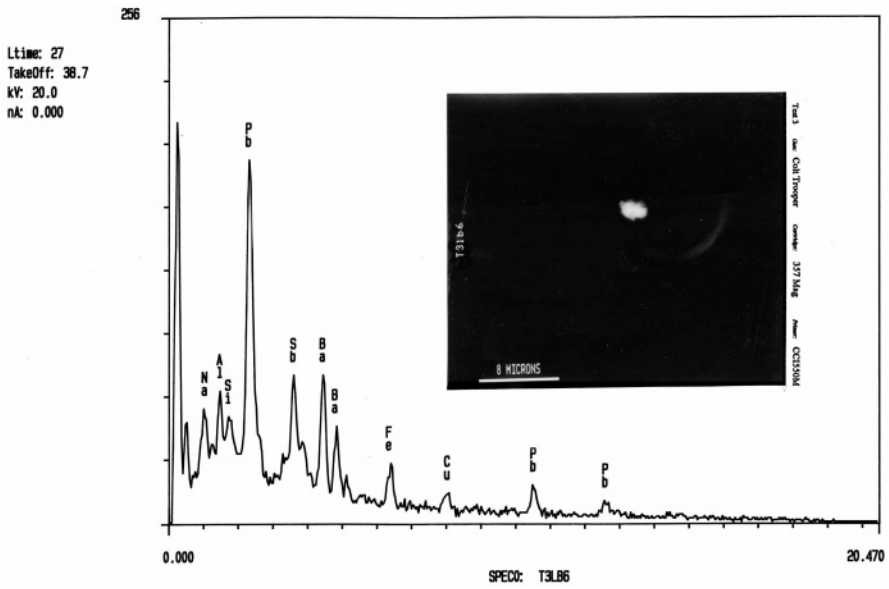


Figure 5.18 Colt Trooper 357 MAG CCI.

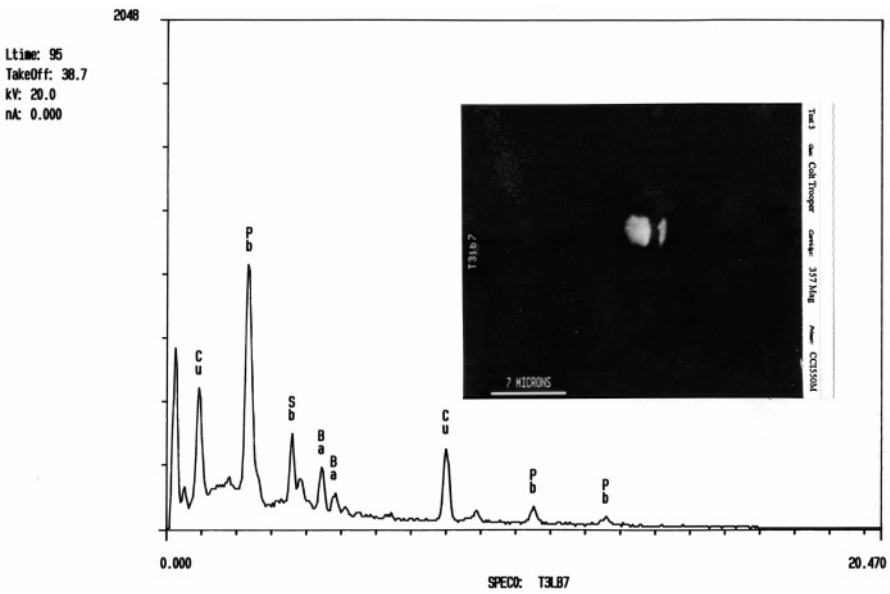


Figure 5.19 Colt Trooper 357 MAG CCI.

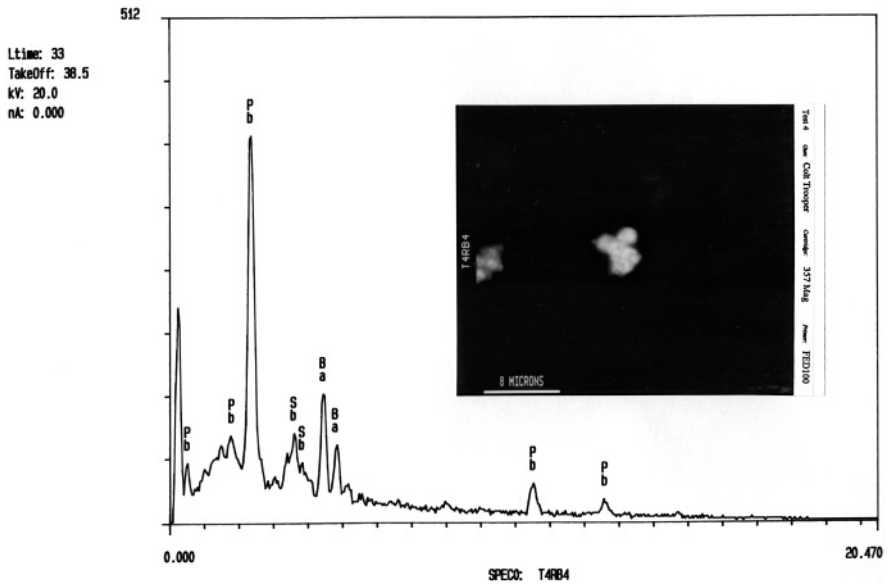


Figure 5.20 Colt Trooper 357 MAG FED.

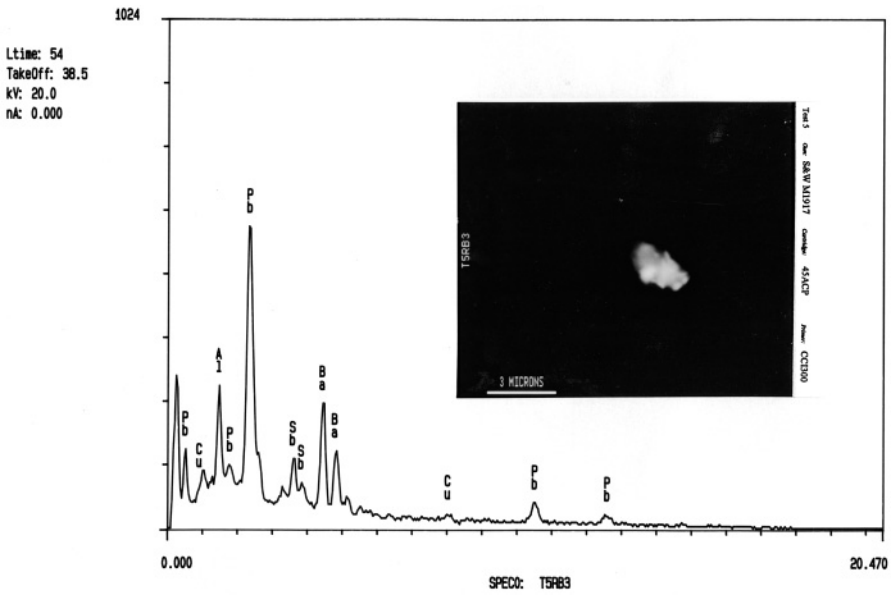


Figure 5.21 S&W MOD 1917 45 ACP CCI.

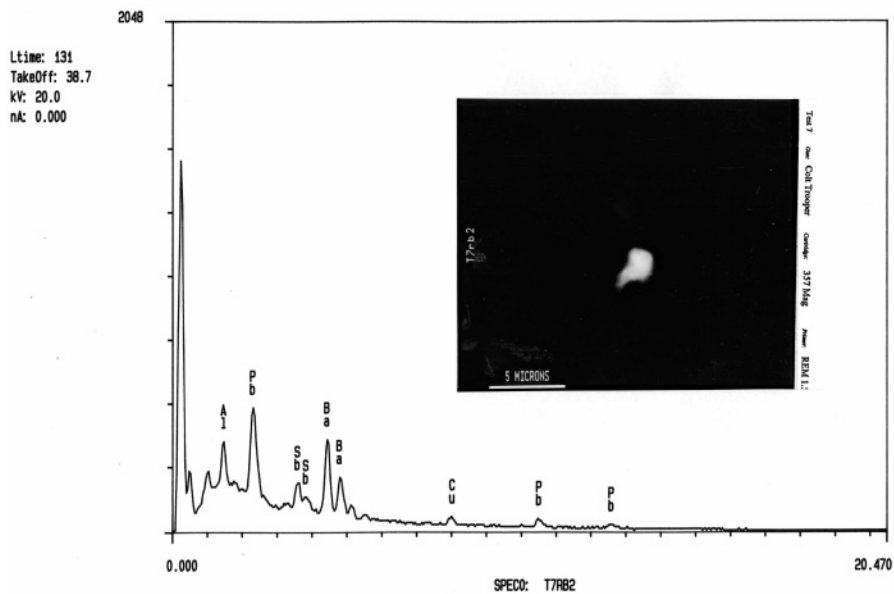


Figure 5.22 Colt Trooper 357 MAG REM.

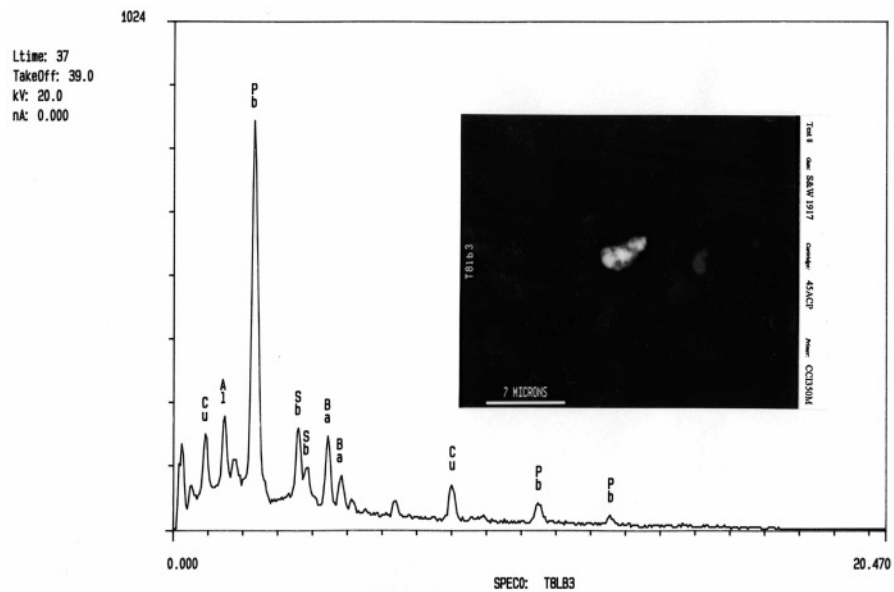


Figure 5.23 S&W MOD 1917 45 ACP CCI.

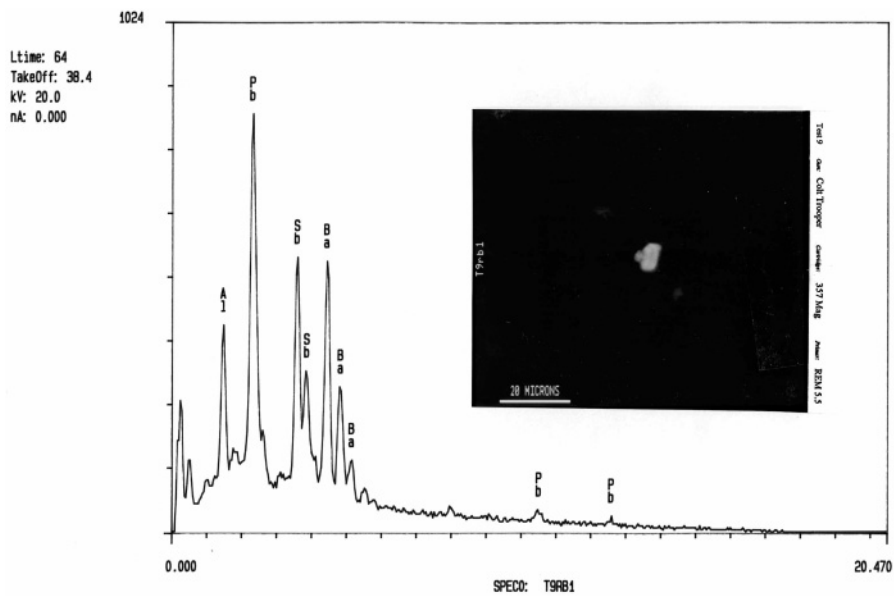


Figure 5.24 Colt Trooper 357 MAG REM.

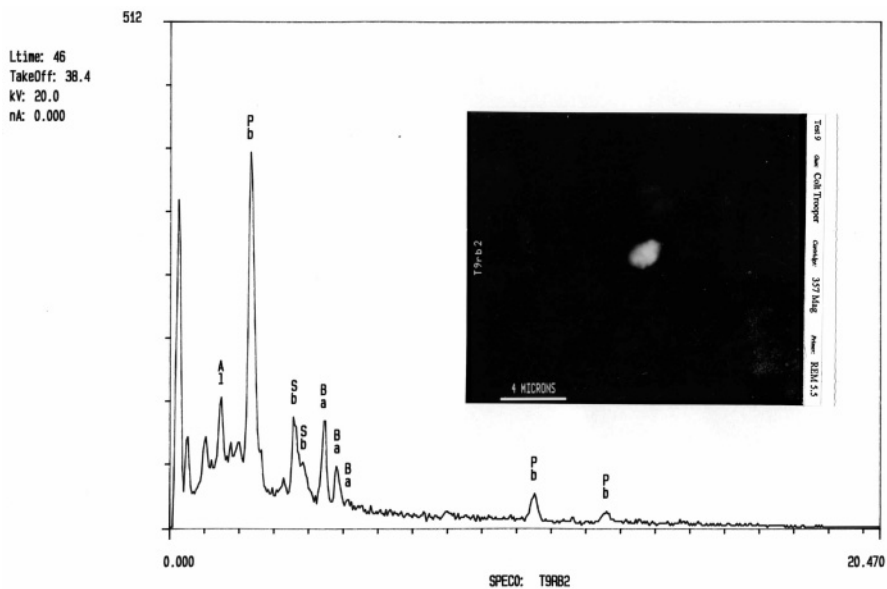


Figure 5.25 Colt Trooper 357 MAG REM.

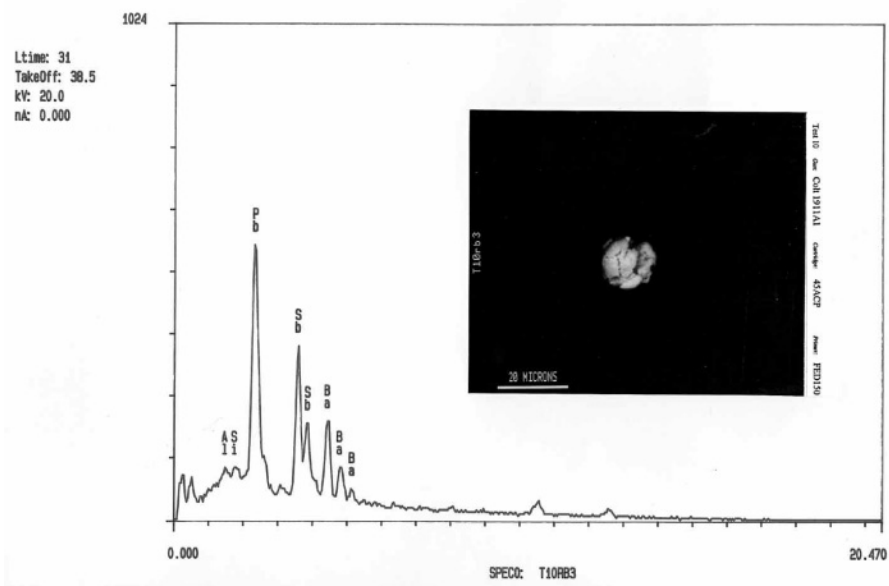


Figure 5.26 Colt MOD 1911A1 45 ACP FED.

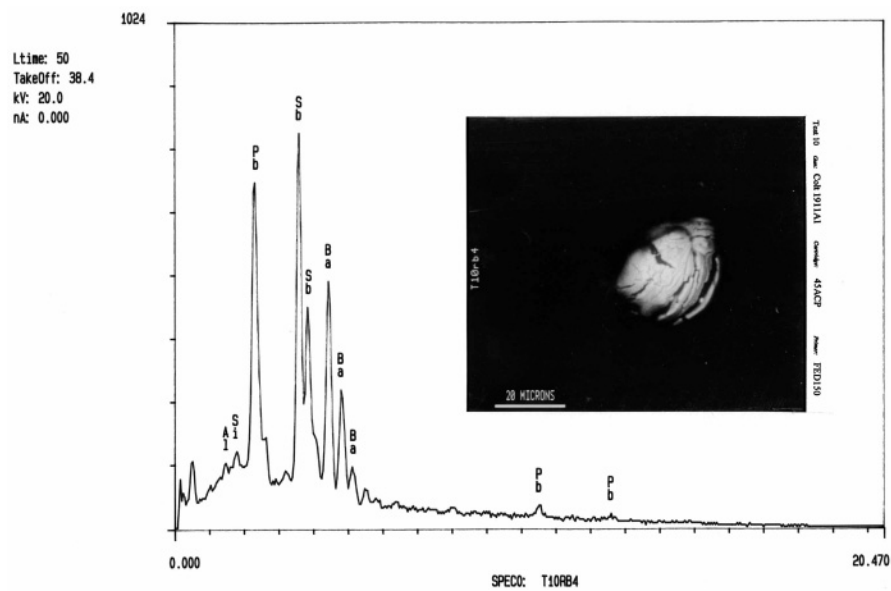


Figure 5.27 Colt MOD 1911A1 45 ACP FED.

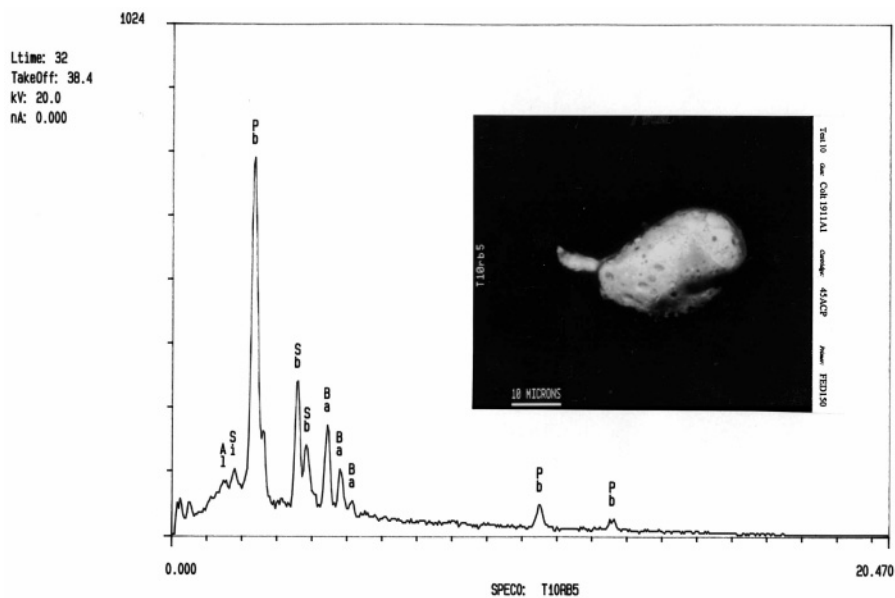


Figure 5.28 Colt MOD 1911A1 45 ACP FED.

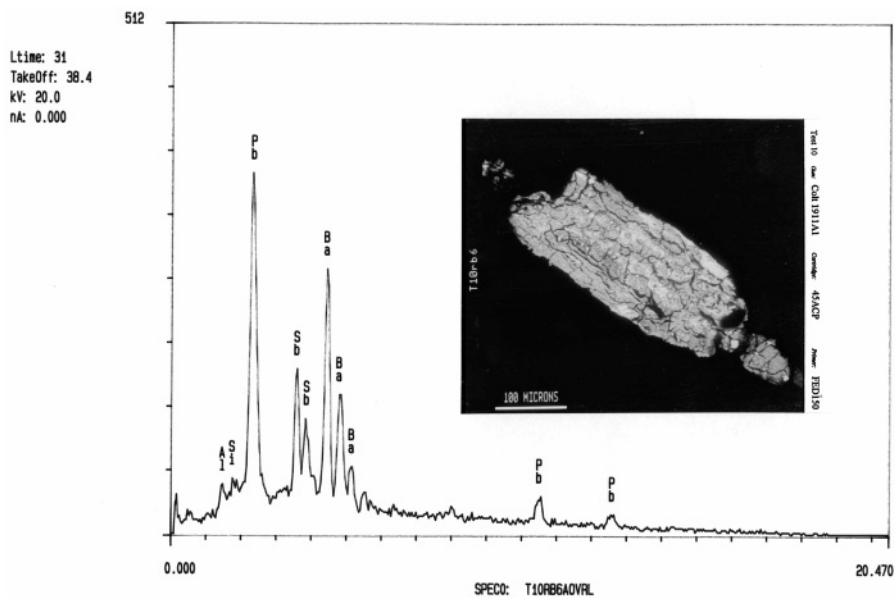


Figure 5.29 Colt MOD 1911A1 45 ACP FED.

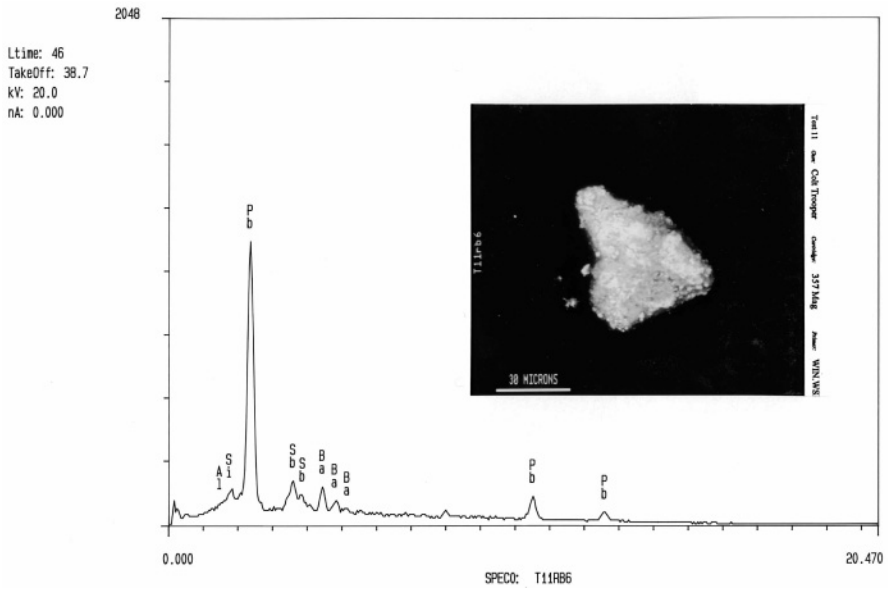


Figure 5.30 Colt Trooper 357 MAG WIN.

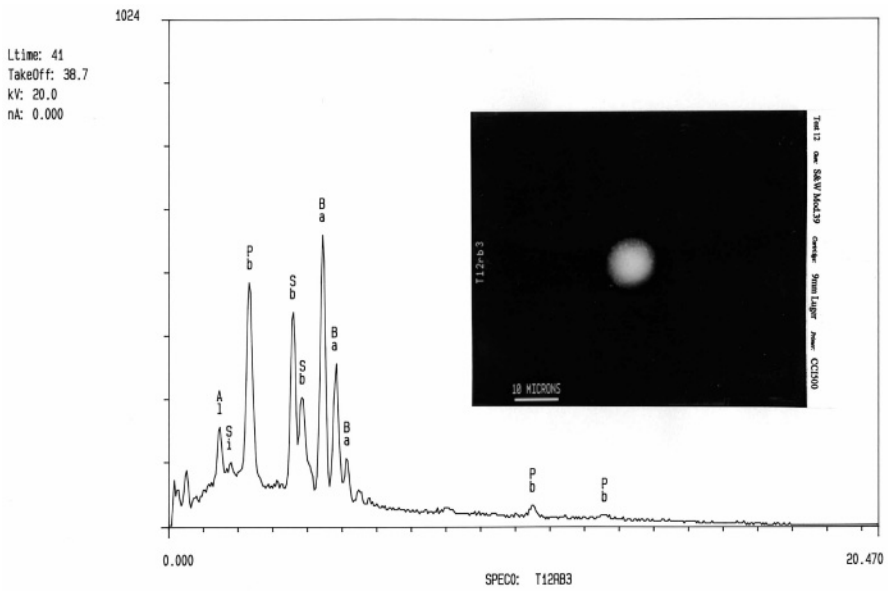


Figure 5.31 S&W MOD 39 9mm Luger CCI.

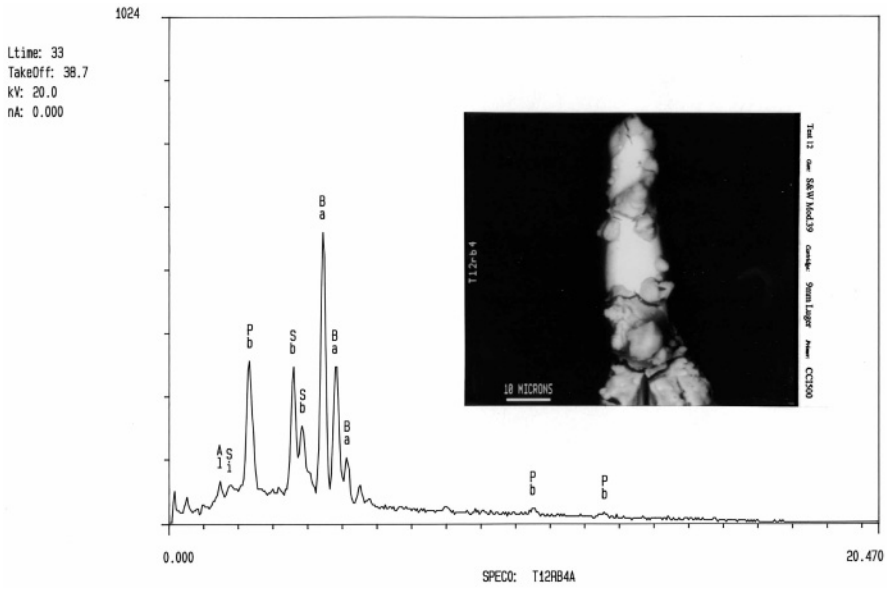


Figure 5.32 S&W MOD 39 9mm Luger CCI.

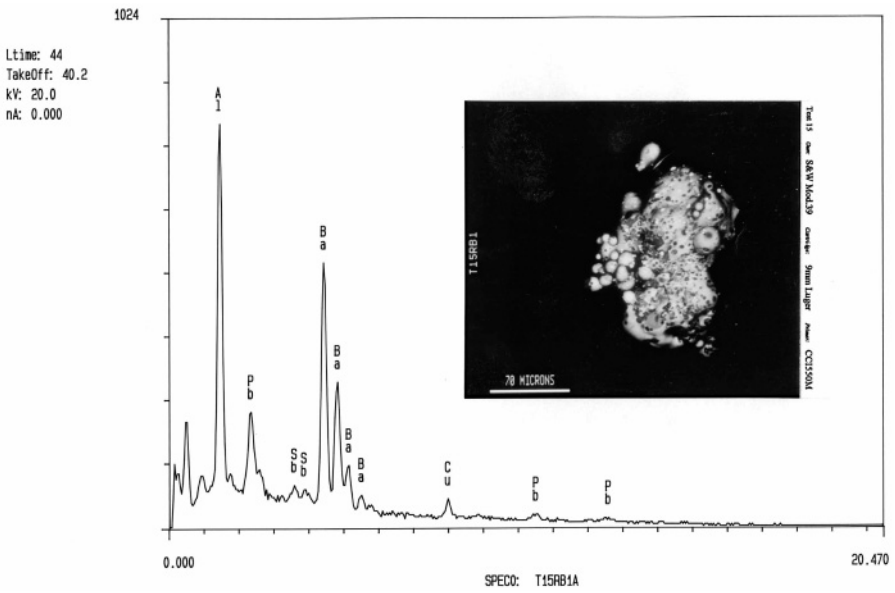


Figure 5.33 S&W MOD 39 9mm Luger CCI.

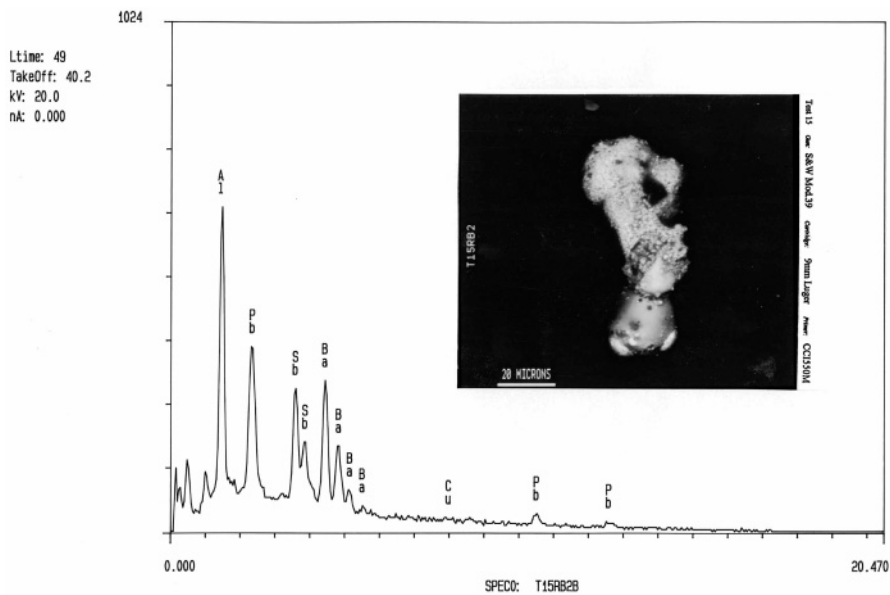


Figure 5.34 S&W MOD 39 9mm Luger CCI.

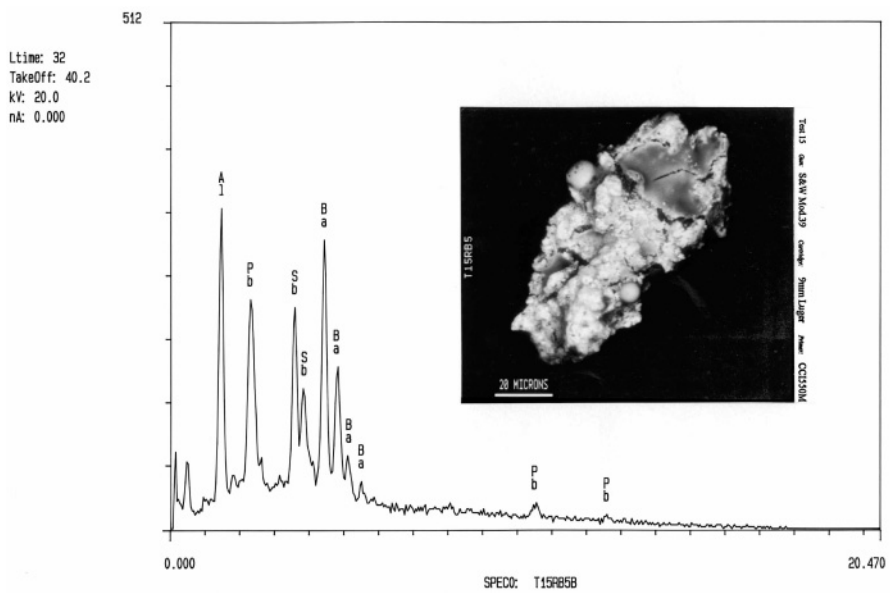


Figure 5.35 S&W MOD 39 9mm Luger CCI.

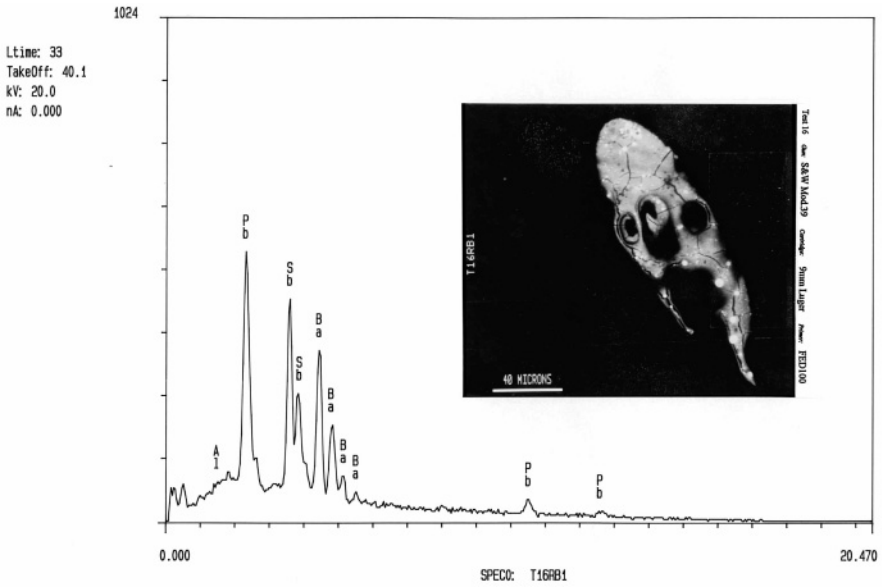


Figure 5.36 S&W MOD 39 9mm Luger FED.

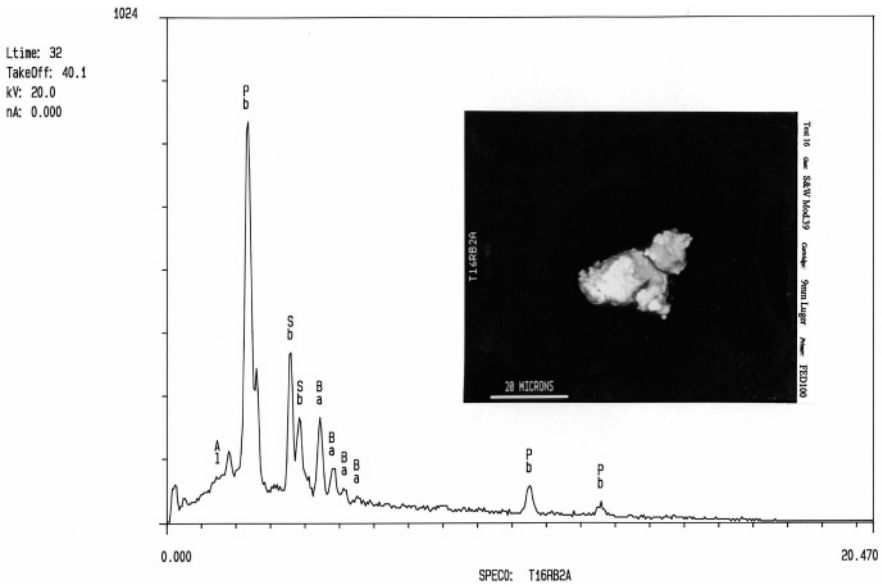


Figure 5.37 S&W MOD 39 9mm Luger FED.

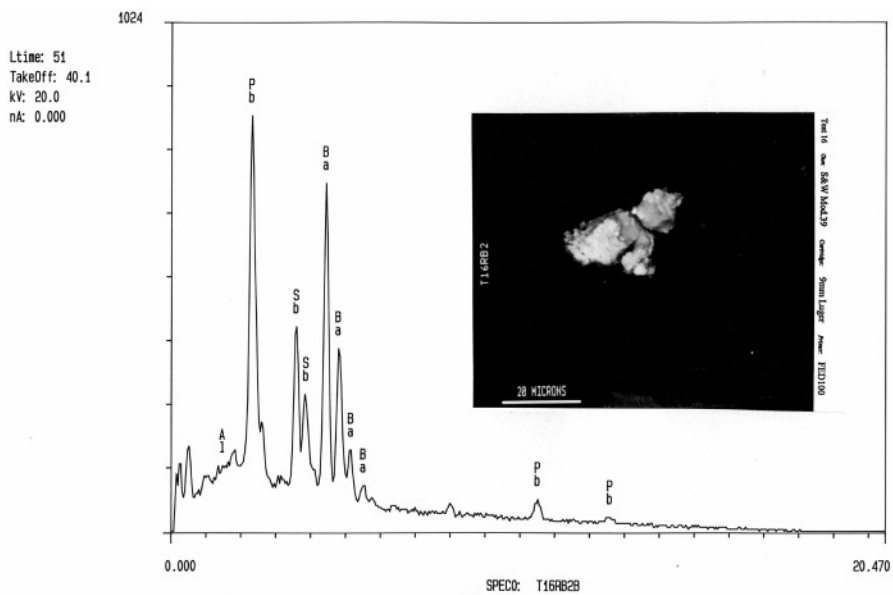


Figure 5.38 S&W MOD 39 9mm Luger FED.

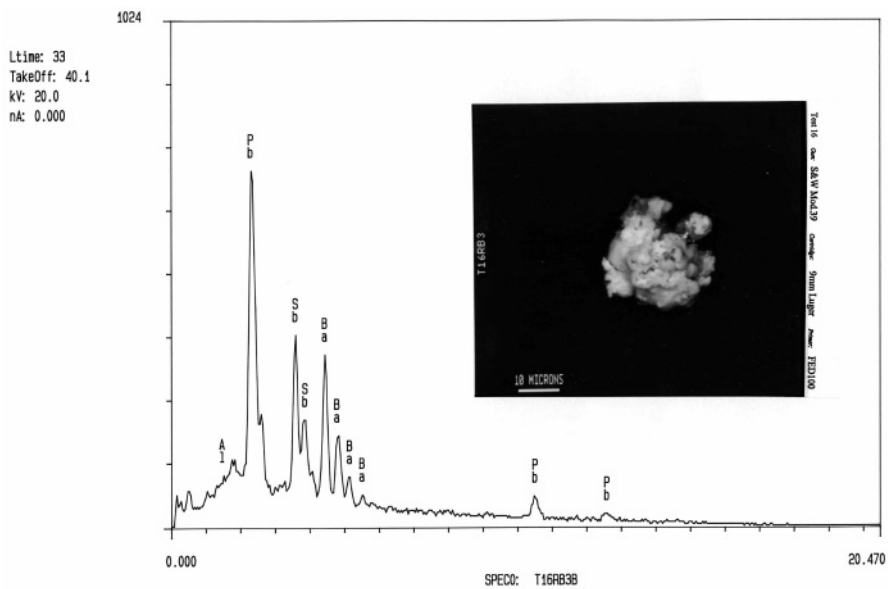


Figure 5.39 S&W MOD 39 9mm Luger FED.

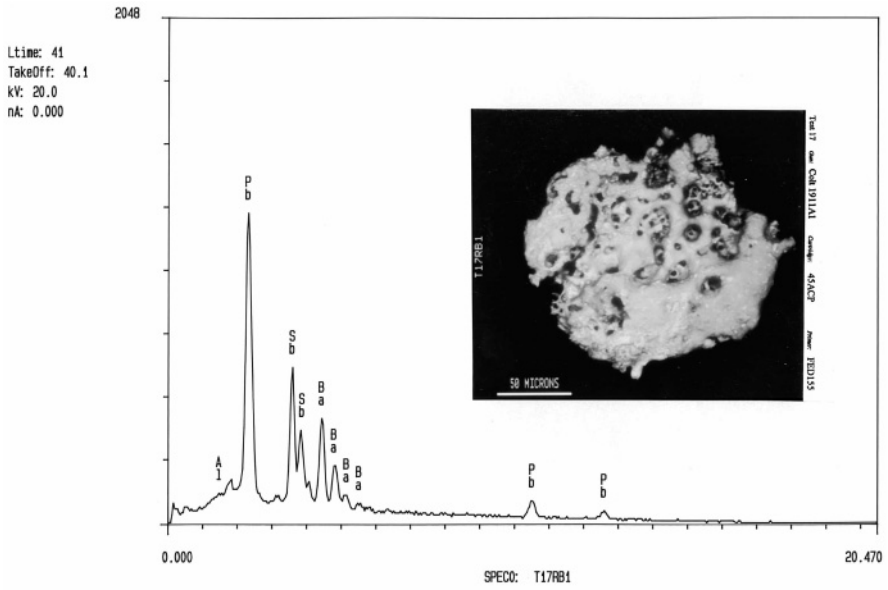


Figure 5.40 Colt MOD 1911A1 45 ACP FED.

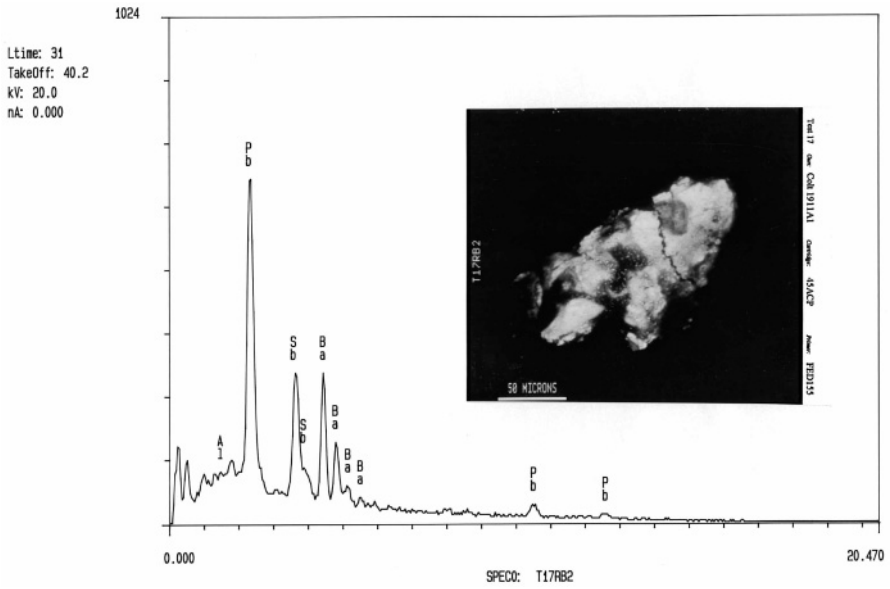


Figure 5.41 Colt MOD 1911A1 45 ACP FED.

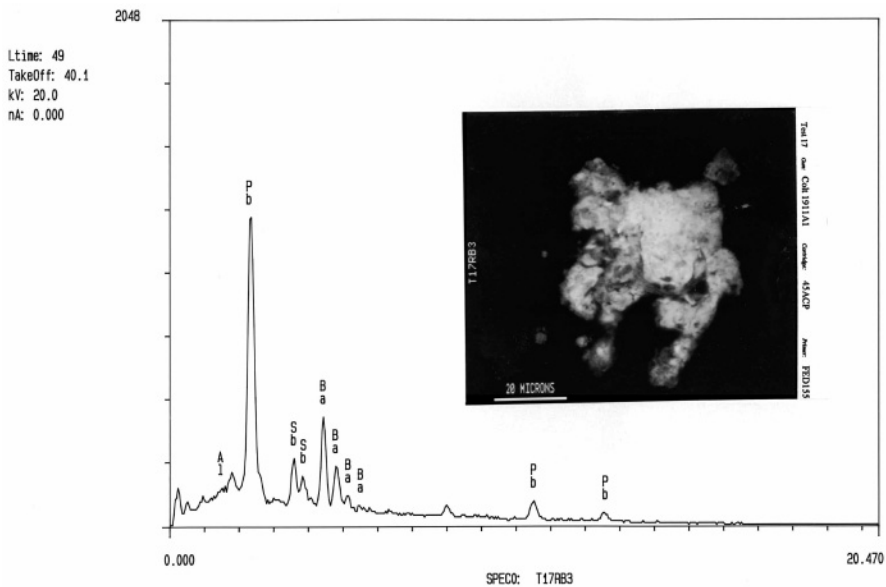


Figure 5.42 Colt MOD 1911A1 45 ACP FED.

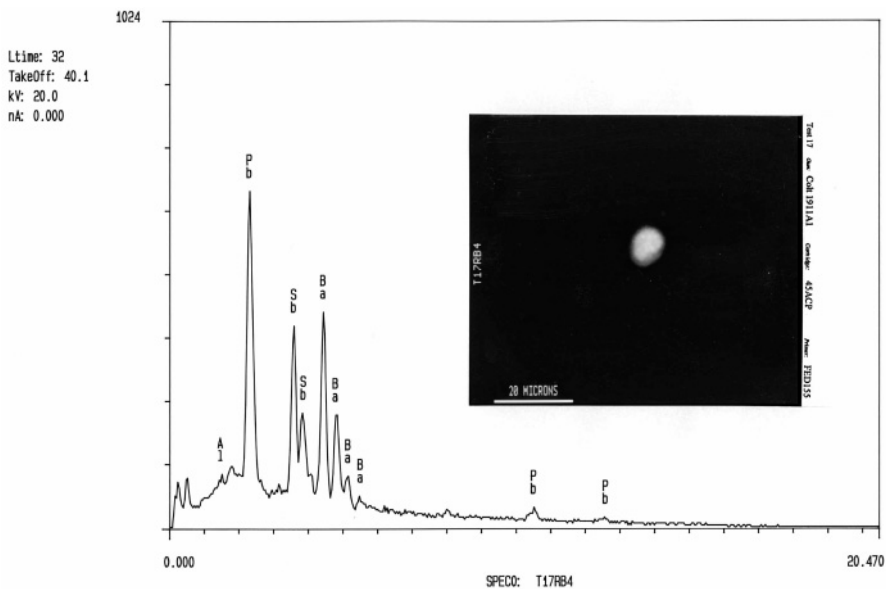


Figure 5.43 Colt MOD 1911A1 45 ACP FED.

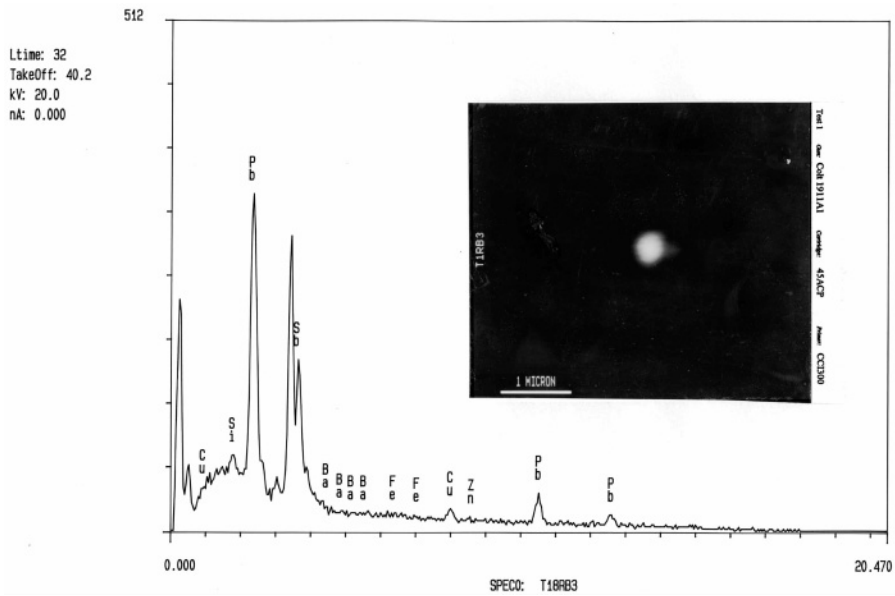


Figure 5.44 Colt MOD 1911A1 45 ACP FED.

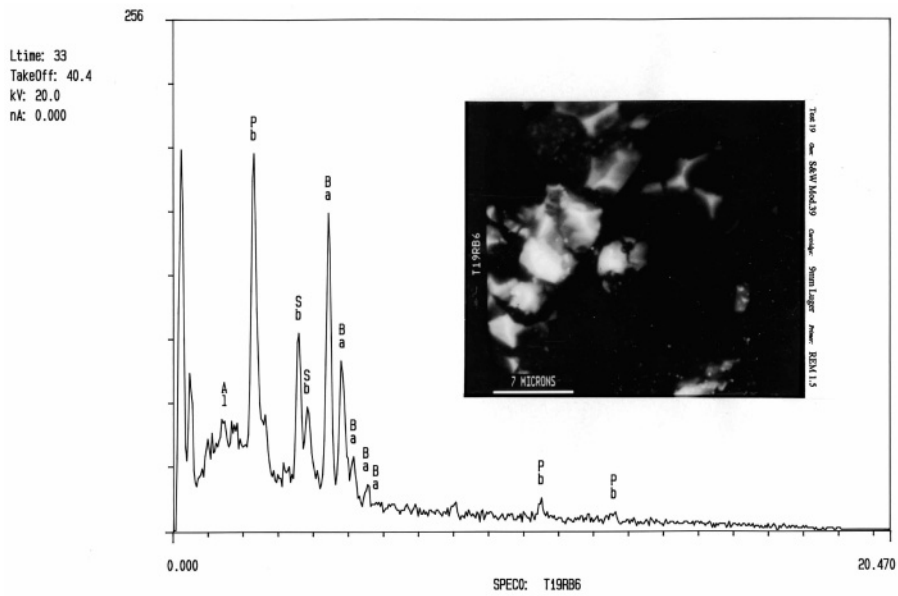


Figure 5.45 S&W MOD 39 9mm Luger REM.

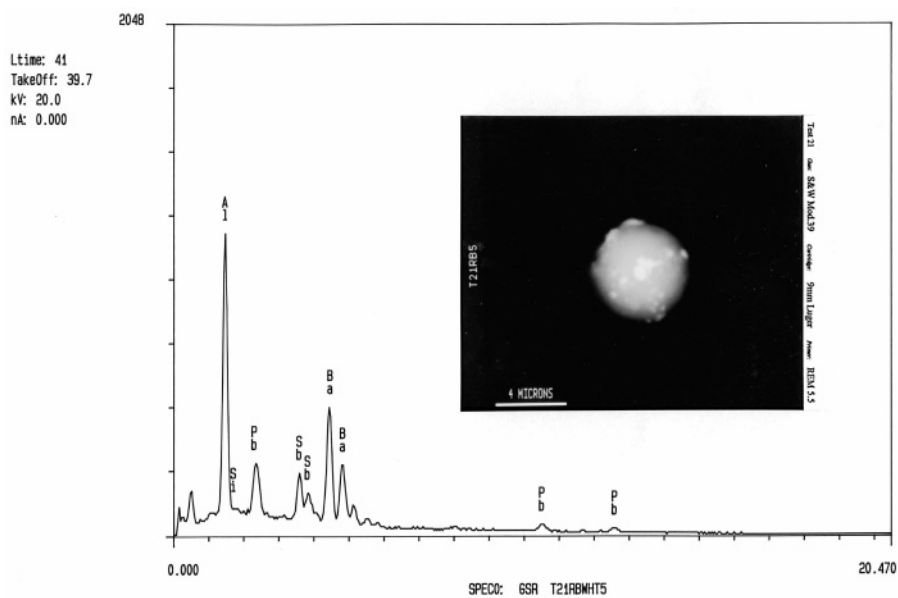


Figure 5.46 S&W MOD 39 9mm Luger REM.

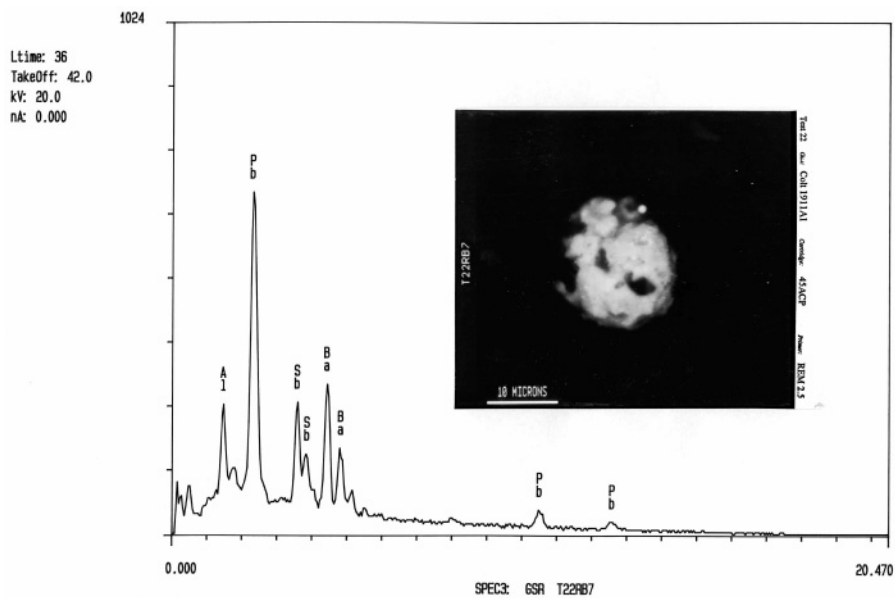


Figure 5.47 Colt MOD 1911A1 45 ACP REM.

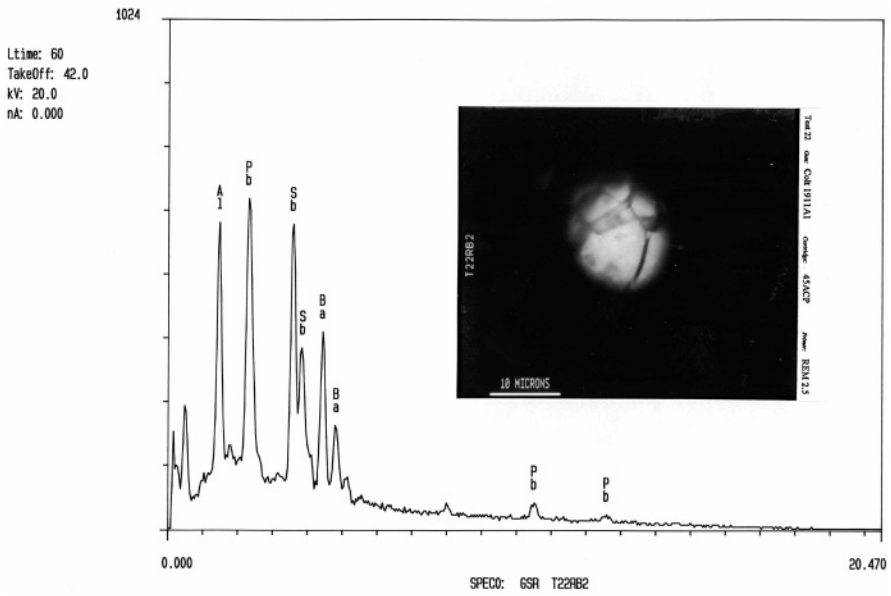


Figure 5.48 Colt MOD 1911A1 45 ACP REM.

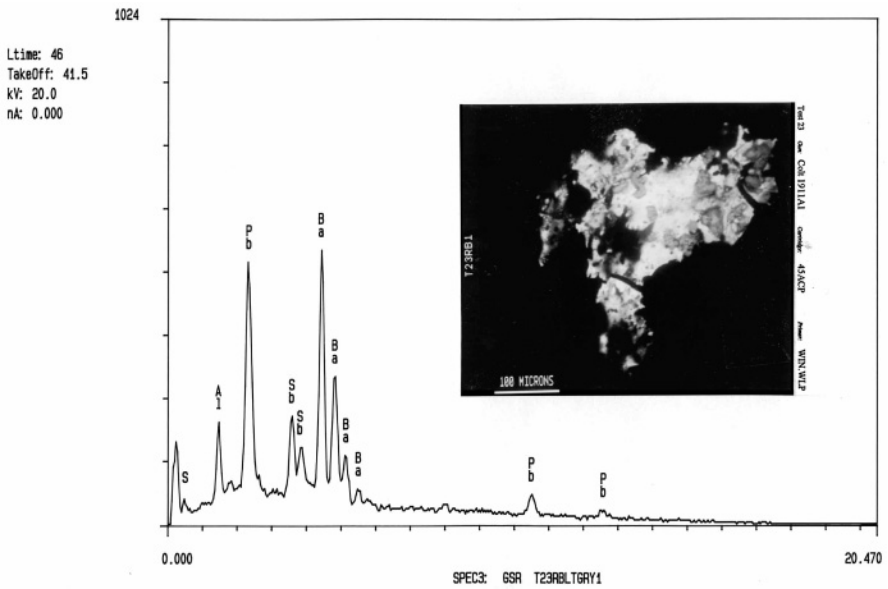


Figure 5.49 Colt MOD 1911A1 45 ACP WIN.

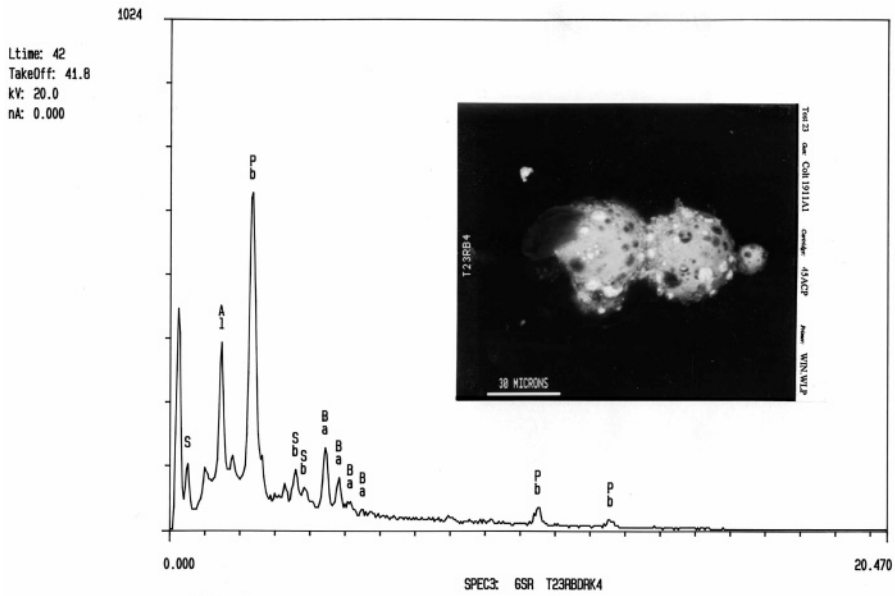


Figure 5.50 Colt MOD 1911A1 45 ACP WIN.

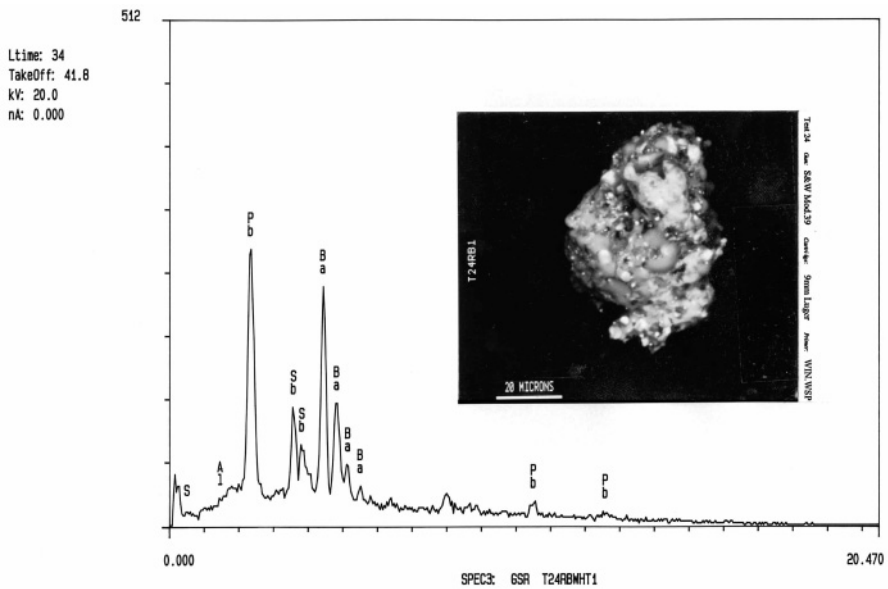


Figure 5.51 S&W MOD 39 9mm Luger WIN.

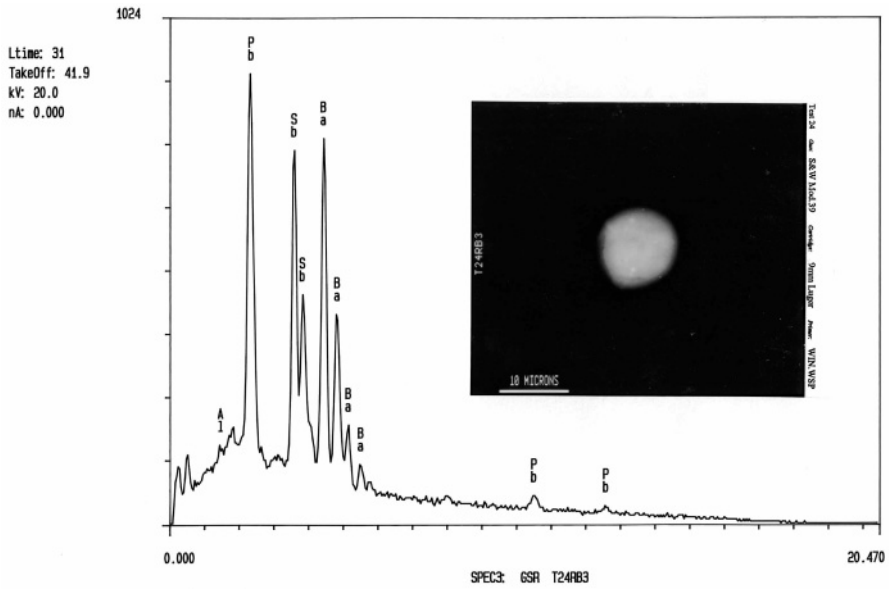


Figure 5.52 S&W MOD 39 9mm Luger WIN.

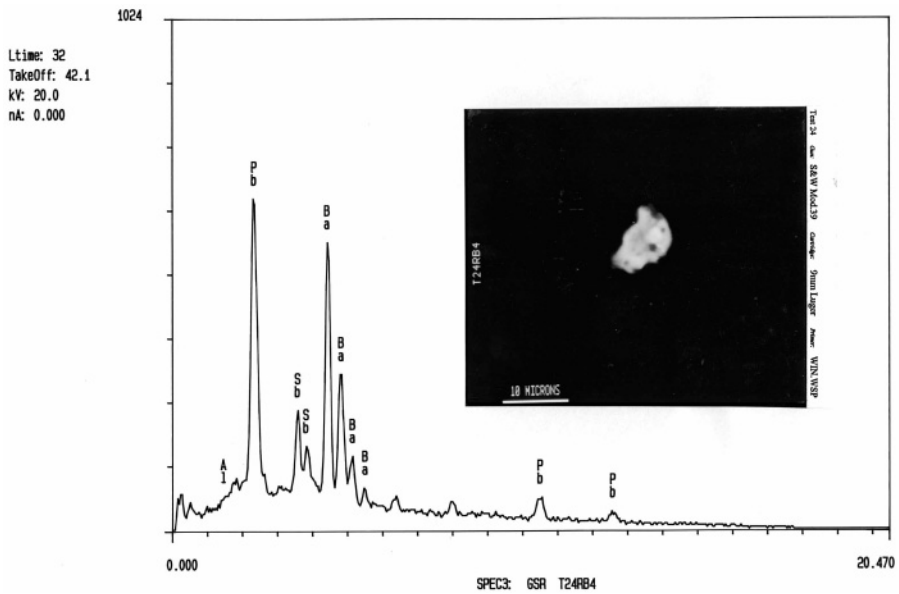


Figure 5.53 S&W MOD 39 9mm Luger WIN.

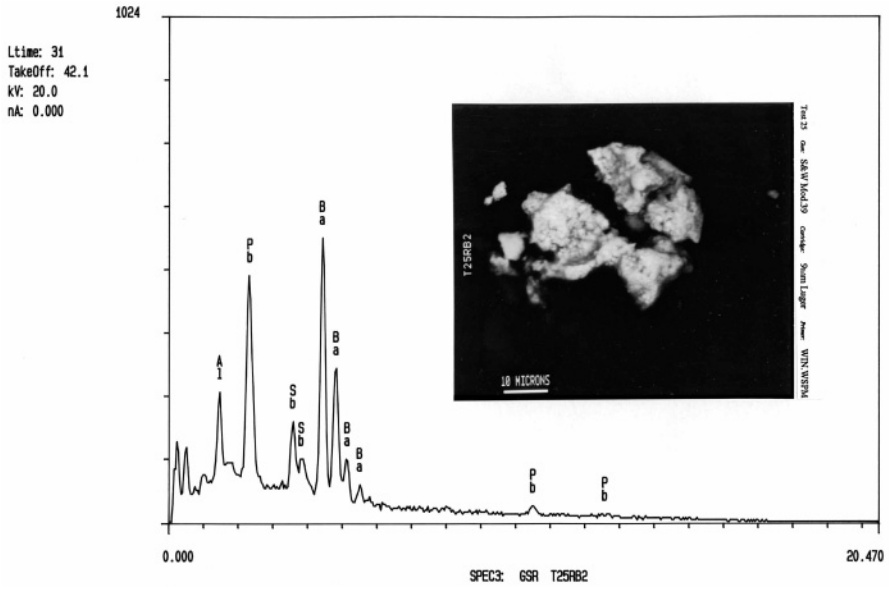


Figure 5.54 S&W MOD 39 9mm Luger WIN.

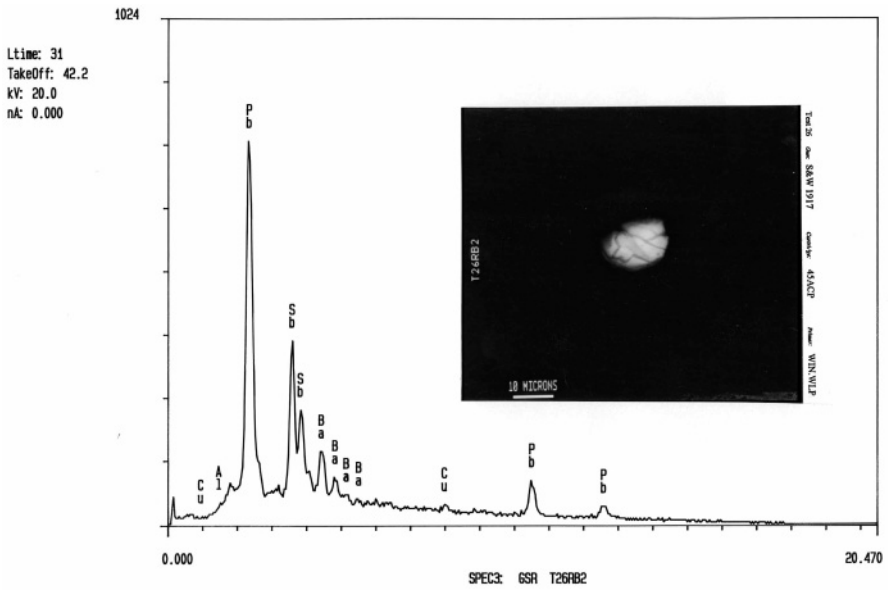


Figure 5.55 S&W MOD 1917 45 ACP WIN.

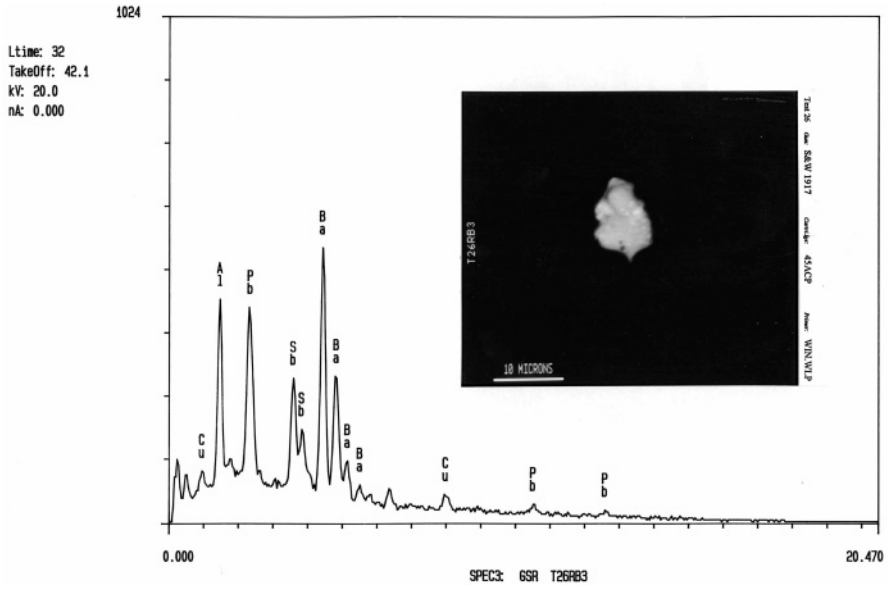


Figure 5.56 S&W MOD 1917 45 ACP WIN.

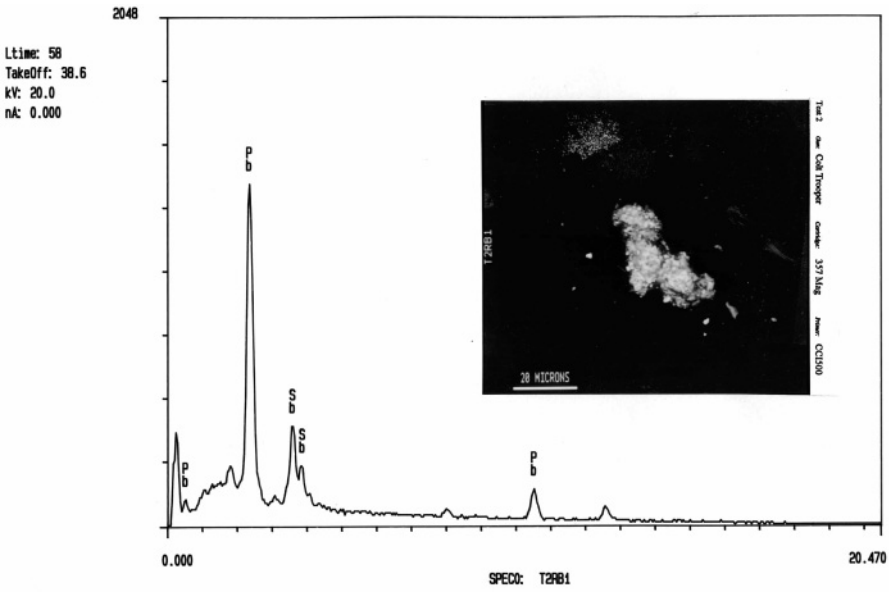


Figure 5.57 Colt Trooper 357 MAG CCI.

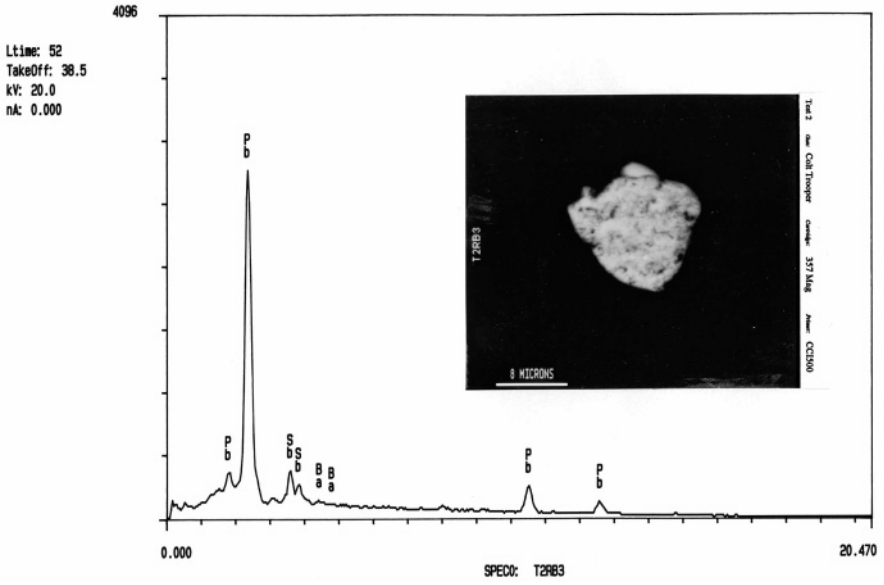


Figure 5.58 Colt Trooper 357 MAG CCI.

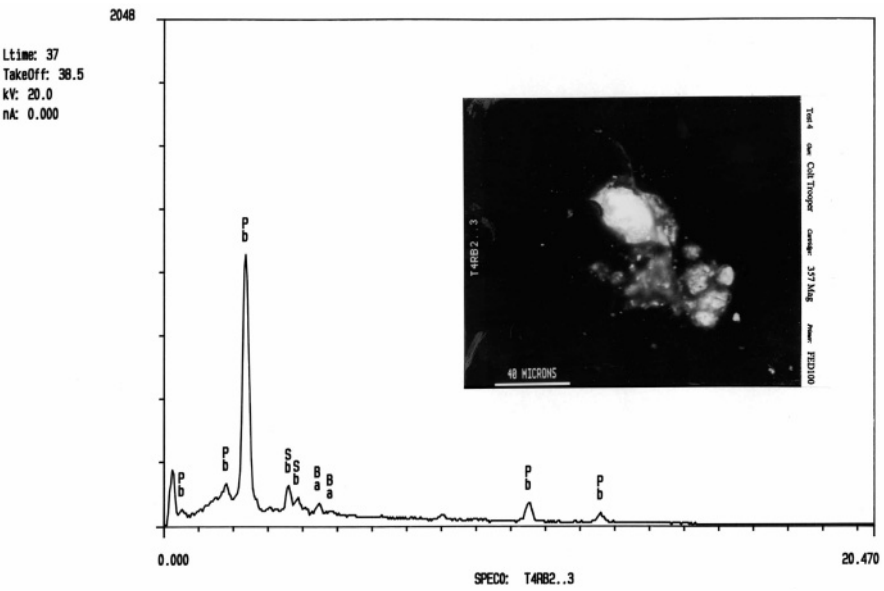


Figure 5.59 Colt Trooper 357 MAG FED.

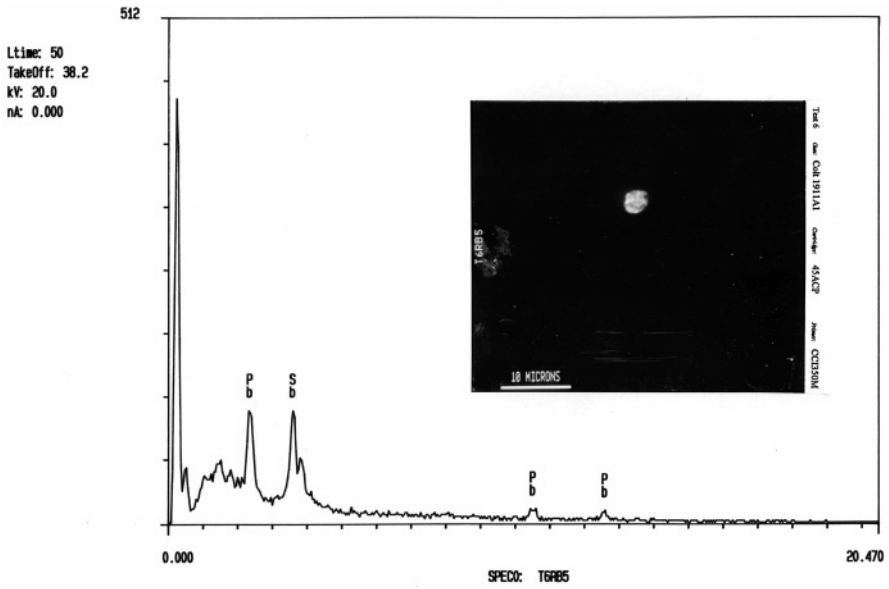


Figure 5.60 Colt MOD 1911A1 45 ACP CCI.

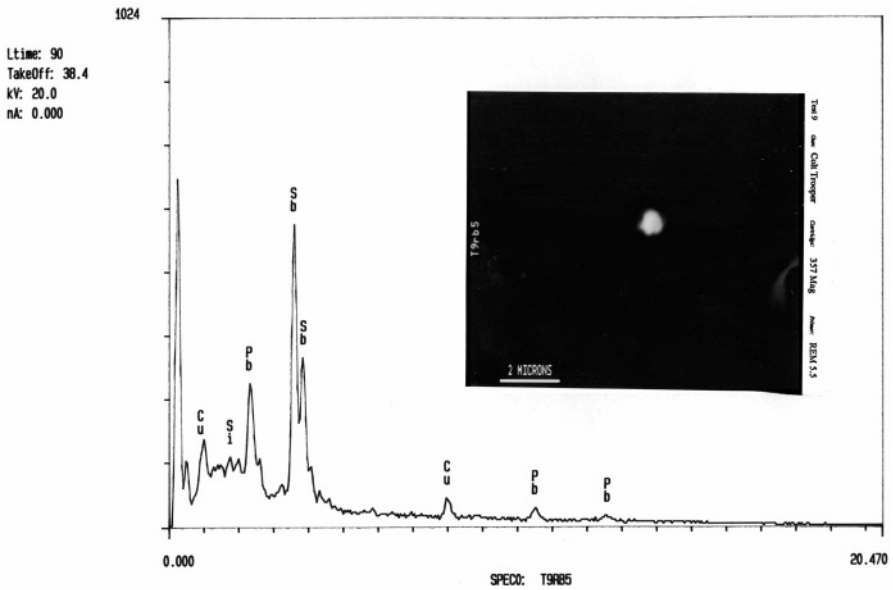


Figure 5.61 Colt Trooper 357 MAG REM.

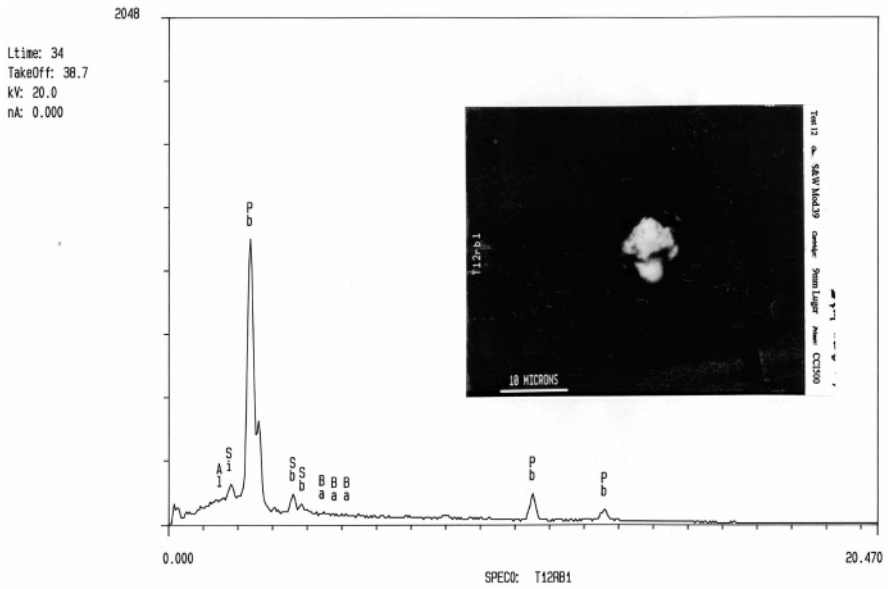


Figure 5.62 S&W MOD 39 9mm Luger CCI.

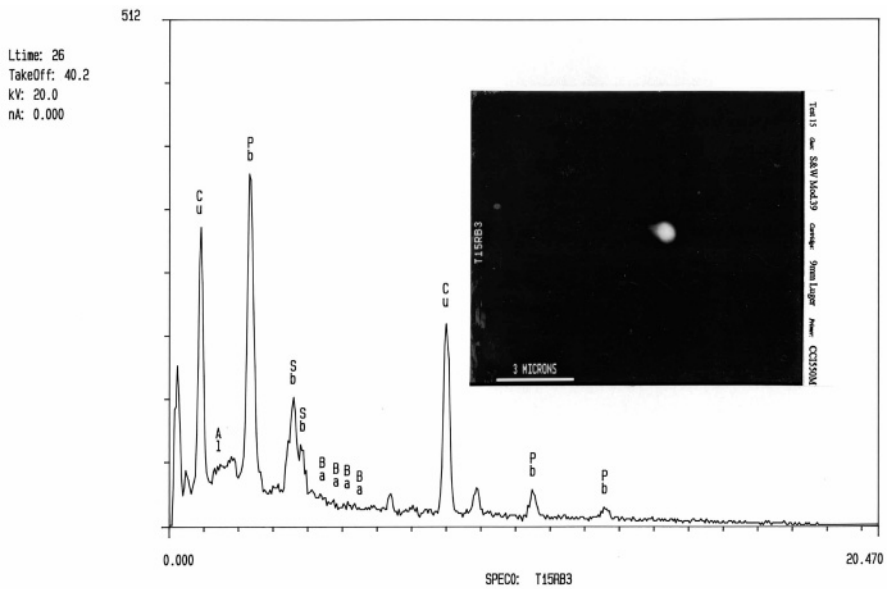


Figure 5.63 S&W MOD 39 9mm Luger CCI.

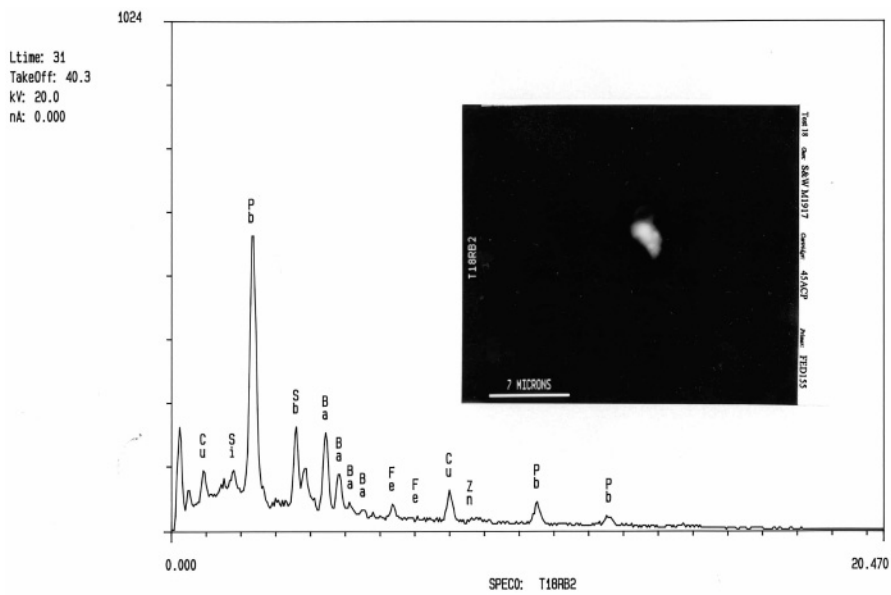


Figure 5.64 S&W MOD 1917 45 ACP FED.

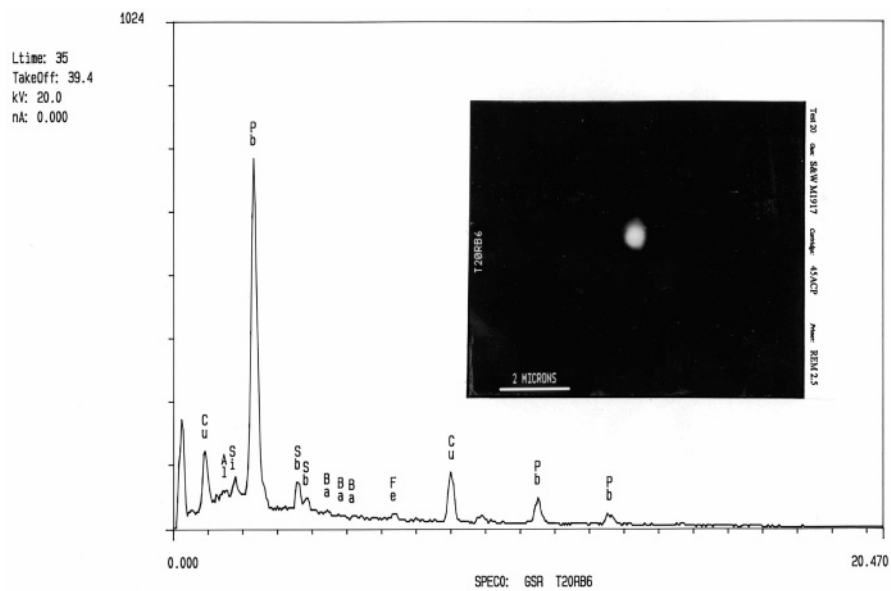


Figure 5.65 S&W MOD 1917 45 ACP REM.

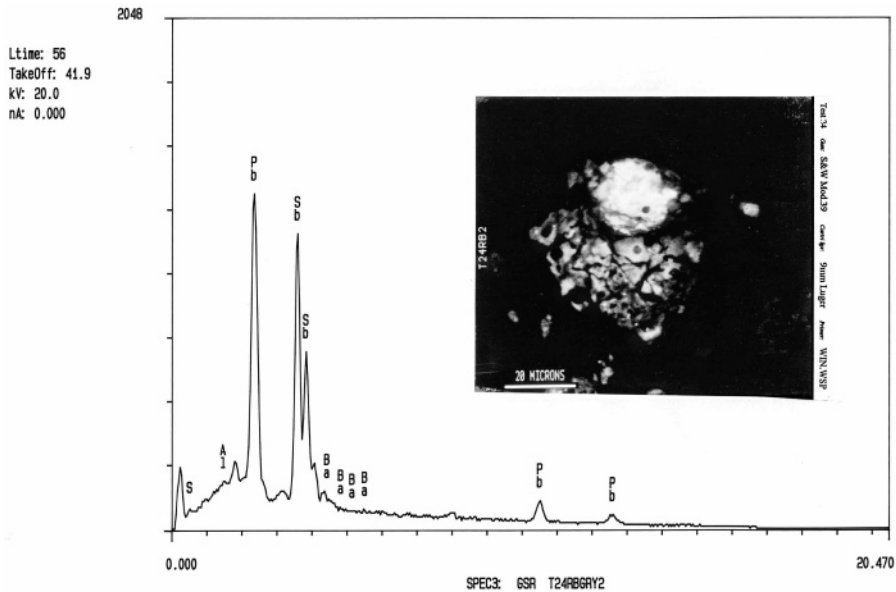


Figure 5.66 S&W MOD 39 9mm Luger WIN.

Revolvers vs. Semi-Automatics

Examination of different primer compositions by collection and analysis of GSR particles resulting from the discharge of a firearm for the purpose of identifying the ammunition manufacturer was once a promising study, producing data that actually appeared meaningful. When outliers became too frequent in a series of test firings, the data took on a whole new meaning. There were several scenarios about why this was a fruitless program to pursue. The most disturbing scenario was when a manufacturer without enough primer on hand to fill a very large order acquired primers from other manufacturers. Since there is no way to identify those separate lots, a large population of outliers resulted. Because of this scenario, this avenue of GSR identification was abandoned, at least temporarily. Looking at the data from a different perspective, we explored the possibilities of other differences in the GSR particulate.

Particle size distribution presents an interesting trend when weapon types are considered. The paths that gases and forming particulate take upon escape from a revolver vs. a semi-automatic weapon create different conditions during the formation of the particles. In the revolver, most of the gases from the primer burn escape in the area around the cylinder, whereas in the semi-automatic, the initial path is the opening provided by the ejection port. These observations were confirmed in the plume study video as described in [Chapter 4](#). A noticeable difference was apparent in the particle size distributions from the two

Table 5.2 Average Particle Size of Particles Generated from the Discharge of Revolvers and Semi-Automatic Weapons

	<3 μ m	<6 μ m	>10 μ m
REVOLVERS	66%	93%	2%
SEMI-AUTOS	47%	76%	14%

weapon types in our preliminary data. Revolvers appeared to generate more smaller particles than semi-automatic weapons, whereas semi-automatic weapons appeared to generate larger particles, as Table 5.2 shows.

Sample collection in this series of tests was taken immediately after weapon discharge. Particle size data represent the total population of possible particle size ranges before any physical activity of the shooter. Further testing, under controlled conditions, with samples taken after limited physical activity, will be completed as time and funds become available. The missing piece of the puzzle may be the influence of physical activity and its effect on the particle size distributions for the different weapon types. These limited observations only involve selected handguns. Further studies about using other weapon types and ammunitions must be conducted before any significance can be given to this preliminary data.

References

1. Frost, G. E., *Ammunition Making — An Insider's Story*, National Rifle Association, Washington, D.C., May 1990, 47–69.
2. Wrobel, H. A., Millar, J. J., and Kijek, M., Identification of ammunition from gunshot residue and other cartridge related materials — a preliminary model using .22 caliber rimfire ammunition, *J. Forensic Sci.* 43 (2), 324, 1998.
3. Andrews, D., *Speer Reloading Manual No. 11*, Omark Industries, Inc., Lewiston, ID, 1987.
4. Waters, D., *Pet Loads*, 3rd ed., Wolfe Publishing Co., Prescott, AZ, 1980.
5. Rammage, C., Kenneth, *Lyman's Reloading Handbook*, 46th ed., Lyman Publications, Middlefield, CT, 1982.

Documentation of Evidence and Testimony

6

Note Taking

Note taking is an essential part of any forensic examination. When evidence is submitted to the laboratory, all relevant information must be documented. This keeps the examination of the evidence orderly, allows evaluation of the testing methods used, and enables the examiner to recall thoughts and procedures when the time comes to communicate to the trier of fact the complete examination of the evidence.

When the evidence is received in the laboratory, the examiner should note who submitted the evidence and the date and time received. This information is also included on the chain of custody form, which should be completed upon initial receipt of the evidence.

Notes should include the case number, date, and initials of examiner. For convenience, they are usually placed in the top right-hand corner of the notes.

Notes vary, depending on the types of analysis to be conducted. For example, for automated gunshot residue (GSR) analysis, a set of printed image sheets is produced that depict the various particles detected. Handwritten notes are also valuable for future recall of information. Notes should include how the item of evidence is packaged and a description of any writing present on the packaging. In many instances, this may be accomplished by photocopying the packaging. The examiner's thoughts are also recorded to refresh the examiner regarding what his or her observations were when the items were examined.

Pictures may be drawn to depict areas of clothing or items and to remind the examiner where some collection or defect was present. Remember that a picture is worth a thousand words. Page numbers should be written in the lower right-hand corners pages of the report.

Pre-designed handouts are also commonly used when manual analyses are conducted. Pre-designed handouts also allow the examiner to simply fill in the areas covered in the analysis and to choose predetermined conclusions.

Report Writing

When a GSR examination is complete, it is essential that the examiner communicate the results of the examination to the proper persons involved in the subsequent investigation. This can be accomplished by verbal communication, but all tests and description of the methods should be written in a formal report. The report should include, but not be limited to, the following:

Agency Name

Subject Names which include suspects and victims involved in the crime

Date of Report

Agency Case Number

Internal Laboratory Case Number

The items of evidence should be described:

Each item number should be placed in the report.

The types of examination that were conducted:

The type of examination should be specified in the report for various reasons. The most obvious reason is that different types of analysis may give more or less specific information than another.

Results of examination for each item should be specified.

This allows the reader and the examiner to categorize the results in a clear and concise fashion. For example, if unique GSR particles were detected on the right back area of a suspected shooter and no other particles were detected on the right palm, left back, and left palm, this would be expected in a right-handed person who has fired a gun. But if high numbers of particles are present on the palms and are absent on the back of the hands, this would lead one to assume that a firearm was handled, but may not have been fired by that individual.

A section covering the conclusions of the analysis are the most critical part of the report. This is where the examiner conveys his or her opinion of the results of the examination. Typical conclusions can be:

For the presence of *unique* GSR particles:

Unique GSR particles were detected on Sample A. These particles may have originated from the discharge of a firearm, being in close proximity to a discharging firearm, or from the handling of a contaminated firearm and/or ammunition components.

For the presence of *characteristic* GSR particles:

Characteristic GSR particles were detected on Sample A. These particles may have originated from the discharge of a firearm, being in close proximity to a discharging firearm, from the handling of a contaminated firearm and/or ammunition components, or from some other source.

This is an important point. What has been discussed in several other areas of this book — that particles that have the same composition and morphology of characteristic GSR particles (Sb, Ba, Pb-Sb, Pb-Ba, Sb-Ba, Sb-Ba-Ca-Trace S) — has been observed in other types of samples.

When no GSR-related particles are detected in a sample:

No unique or characteristic GSR particles were detected on any of the submitted samples. Accordingly, the results of the examination are termed inconclusive.

The results are termed inconclusive for many reasons. First and foremost, the activity of the subject may be unknown. Also, studies show that the persistence of GSR particles decreases over time. If a subject has washed or cleaned the hands in some manner, the likelihood of detecting GSR decreases. The hands may have been covered with gloves or some other covering. For these reasons and many others, negative results obtained from an examination are not necessarily exculpatory.

The signature and title of the examiner should conclude a report. This allows the reader to see who conducted the analysis. All reports should be reviewed by a second examiner for two reasons: first and foremost, to ensure that all the conclusions are clear and concise and that the results are complete, and second, administrative review should be conducted to ensure that item numbers and spelling are accurate. An error in spelling or in recording the proper item number may lead to great confusion later.

An example of a GSR report follows. The report can be modified, and information may be added or deleted depending on the policy of the laboratory.

Sample SEM Report for Gunshot Residue Analysis

Introduction

_____ sample was received from

_____ of _____ for GSR (gunshot residue) analysis and particle extraction samples were collected from the item(s) at our laboratory using permanently tacky adhesive stubs. The samples were identified as follows:

OR

_____ gunshot residue (GSR) kit containing _____ samples was received from _____ for GSR analysis. The samples were identified as follows:

Agency Sample ID	Sample No.
Case No.	
Right Back	1001
Right Palm	1002
Left Back	1003
Left Palm	1004
Control	1005

The particle extraction samples were placed directly into the PSEM (PERSONAL SEM®) for analysis without any further sample preparation.

SEM ANALYSIS

The samples were examined initially using manual microscopy to set run parameters and sample analysis area. They were then analyzed using an automated scanning electron microscope (PERSONAL SEM®) equipped with a full gunshot residue analysis package, including automated stage, backscattered electron (BSE) detector, energy dispersive X-ray spectrometer (EDS), and automated GSR analysis software.

When lead, antimony, and barium combine or fuse into a single particle, it is defined as being unique to GSR, since there are no other known sources of this compound. Each individual element or other combinations of these elements with high-temperature features or characteristics are classified as characteristic of GSR.

The SEM analysis, on a particle-by-particle basis, retains the individual feature characteristics and can relate the presence of lead (Pb), antimony (Sb), and barium (Ba) to a single particle. When the instrument detects particles with the presence of Pb, Sb, and Ba, it flags the particles as potential GSR and stores images, composition, and coordinate data for relocation and confirmation by manual microscopy after the automated analysis is completed. A summary run sheet is printed with stored images and spectral data for relocation and confirmation applications. Summary sheets of flagged potential GSR particles from the automated analyses are found in the appendix of this report. All flagged particles were relocated for compositional confirmation.

ANALYTICAL RESULTS

A list of confirmed particles detected during the analysis is as follows:

(LIST FINDINGS.)

CONCLUSIONS

Particles confirmed as unique to GSR could have resulted from the discharge of a firearm, being in close proximity to a discharging firearm, or from the handling of a contaminated firearm and/or ammunition components.

(This conclusion is used when unique gunshot residue particles, i.e., Pb/Ba/Sb are present in the sample.)

AND/OR

Particles confirmed as characteristic of GSR could have resulted from the discharge of a firearm, being in close proximity to a discharging firearm, from the handling of a contaminated firearm and/or ammunition components, or from some other source.

(This conclusion is used when characteristic gunshot residue particles, i.e., Pb/Sb, Pb/Ba, Ba/Sb, Ba, Sb, Ca/Si/Ba/Trace S are present in the sample.)

AND/OR

No particles classified as unique or characteristic to gunshot residue were detected on any of the above submitted samples. Accordingly, the results of this examination are termed inconclusive.

(This conclusion is used when no unique or characteristic gunshot residue particles are detected in the sample.)

Chain of Custody

Chain of custody forms are used in forensic laboratories to track evidence at all times. At any time in the existence of physical evidence, the chain of custody should reflect where, when, and who had possession of the evidence. The main components of a chain of custody form is the name of the agency, case number, items received, a description of the items, and who received the item and when. The form should list and describe all items of evidence and any person who has received or relinquished the particular items of evidence. The name of the person handling the evidence and date and time of receipt or relinquishing of evidence should be filled out each time an item of evidence exchanges hands. The chain of custody is the only way to ensure the integrity of the evidence in question. The fewer people who handle the evidence, the better. The following is an example of a chain of custody form used in RJ Lee Group laboratories.

Chain of Custody

Samples Received From:

Name		
Company		
Mailing Address		
City	State	Zip
Telephone No.	Fax No.	

Client's Sample Identification - Case/Incident No.:
 RJ Lee Group Project No.:

Item	Sample ID	Sample Description
1.		
2.		
3.		
4.		
5.		
6.		
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		

Relinquished by:	Agency:	Date:
<i>Method of Shipment:</i>		
Received by:	Agency:	Date:
<i>Sample Condition Upon Receipt:</i>		
Relinquished by:	Agency:	Date:
<i>Method of Shipment:</i>		
Received by:	Agency:	Date:
<i>Sample Condition Upon Receipt:</i>		

Expert Testimony

General Questions and Possible Explanations

When the GSR expert is called upon to explain the process of GSR analysis, the jury members rely upon this expertise to help them understand what the results of the expert's examination mean. The attorneys must understand GSR and ask the appropriate questions so a complete overview of the methods used to determine the expert's results are reached. The following section is a general description of some common questions and appropriate answers that ensure that everyone in the courtroom understands the significance of GSR evidence.

Expert Qualifications

The first step in any forensic testimony is to qualify the witness as an "expert" in a particular field in which the witness will offer an opinion. This is a critical point because, unlike most witnesses, an expert is permitted to offer an opinion to court based on his or her education, training, and experience.

The attorney should ask the witness questions about background, such as education, and its significance to the witness' current position as a forensic scientist. Another main area that should be explored is the examiner's training in conducting examinations in his or her particular field of expertise. Experience is another area that is important to witness credibility. Questions such as how many cases the expert has conducted or how many samples he or she has examined can give the judge and jury a better understanding of the examiner's experience. Additional questions can add credibility, such as witness publications in the area in which the witness will testify or professional presentations in that area to peers at scientific meetings and symposia. Finally, affiliations to professional organizations can be discussed. When the attorney has gone over the qualifications of the expert, the expert is offered to the court as an expert in that area of forensic science. The sitting judge determines whether the person is qualified as an expert in that particular court.

Background Questions

The first types of questions that should be asked to an expert witness are background questions concerning the type of examination conducted. The following are some general questions and answers that are common in GSR testimony.

Q: What is GSR?

A: In the bottom of the cartridge casing is a round insert called the primer cap. In the primer cap there is a primer compound which is paste material. Primer compound contains lead styphnate, barium nitrate, and antimony sulfide. The lead styphnate is set off by a sharp blow from the firing pin and acts as the initiator, or primary explosive. The barium nitrate increases the heat of reaction and provides oxygen to burn the antimony sulfide, which acts as the fuel with a high burn rate. This reaction ignites the gun powder which is the propellant for the bullet. The heat and pressure generated from the ignition of the primer cause the inorganics of the primer mixture to vaporize. Openings in the gun allow these vapors to escape and condense into droplets of fine particles, which are distributed in the surrounding areas. This can include the hands or clothing of the shooter, the surrounding environment, or others in the vicinity.

Q: What is the plume and approximately how many particles are in a plume?

A: "Plume" is a term used to describe the vapor or cloud that forms around the weapon upon discharge of the firearm. There is no way to determine how many particles are contained in a plume.

Q: What is a unique particle in gunshot residue?

A: An examiner looks at all the particles collected, but the particles that contain the unique combination of Pb-Sb-Ba are unique to gunshot residue. These three elements found together in one particle are unique to GSR. They are found in no other place in the environment.

Q: What are characteristic particles?

A: A characteristic particle is a particle that either has some combination of Pb-Sb-Ba or is made up of one of the elements. However, in the case of unique and characteristic particles the examiner not only considers the elemental composition, but also the morphology and the

entire particle population. Heat-treated particles from the bullet (Pb) or from the casing (Cu/Zn) may also be present.

Q: How many unique and characteristic particles would you expect to find on the hands or clothing of a person suspected of firing a gun?

A: The number of unique and characteristic particles collected on a subject's hand, clothing, etc. depends upon several variables. There is no guarantee that one will find any particles (unique, characteristic, or other) on the hand or clothing of a subject suspected of firing a gun. There are many variables to be considered:

1. The sampling media.
2. The collection technique and efficiency of the collection officer (i.e., was the stub held flat and dabbed over the entire sampling area?)
3. Was the sample collected in a reasonable amount of time after the incident?
4. What was the physical activity of the subject following the questioned shooting and before the collection? (that is, Did the subject wash his or her hands?)
5. If the weapon is unknown, were the highest areas of GSR deposition sampled?

Many other factors may also play a role in the amount of GSR collected. These factors include wind, turbulence, high-speed thoroughfares, drafts caused by open windows/doors/exhaust fans, rain, moving vehicles, and movements of the shooter. Therefore, many factors play a role in the amount of GSR that may be deposited and collected for examination.

Q: Do you consider Pb particles to be characteristic of GSR?

A: The examiner expects to see relatively larger populations of Pb-rich particles to accompany GSR. We consider Pb-rich particles characteristic when they are in the presence of other unique or characteristic gunshot residue particles. Pb-rich particles alone are inconclusive to the examiner because there are so many other environmental sources of Pb. Unique and characteristic particles are normally accompanied by a population of Pb-rich particles. Pb-rich particles by themselves are inconclusive; however, they are reported or noted when found, especially if they are the predominant element, spherical, and appear as heat-treated/molten particles.

Q: What are other sources of Pb?

A: Fuel exhaust is the most common source of Pb and can be characterized as such by the presence of Br. Other sources of Pb include the firing of studguns (construction), smelting of Pb, and manufacturing of plates for lead acid batteries. Electricians and TV repair technicians also may have Pb-Sn or Pb-Sb particles on their hands from solder.

Q: Are Ba and Sb particles found in the environment?

A: Antimony, but no lead or barium, is found in children's cap guns. Other occupational samples have also been shown to contain various amounts of antimony and barium such as exhaust mechanics, brake mechanics, and technicians involved in fireworks displays. It must be pointed out that these occupational particles also contain other elements not typical of GSR. This underscores the experience of the examiner and the examiner's ability to evaluate the other types of particulate present in the sample.

Q: How are the samples examined?

A: The samples were examined using computer-controlled scanning electron microscopy.

Q: How does the scanning electron microscope work?

A: To gain a better understanding of the electron microscope, we will generally compare it to a normal light microscope, which we have all seen in science class or on television. In a light microscope, a light source is sent down a tube, reflects off the surface of the sample, and is sent through a series of lenses to magnify the sample. The image is returned back to the eyepiece. It can then be viewed by looking into the eyepiece of the microscope. In the SEM, the light source is replaced with an electron beam. This electron beam is emitted from a tungsten filament. The beam is focused down the column by a series of electromagnets. The beam interacts with the sample and produces a large number of signals. Some of the electrons that are scattered are collected by a detector to produce an image. This allows the examiner to view the sample at magnifications ranging from 20× to 20,000×. This is much greater than any image that can be viewed with a light microscope. X-ray analysis allows the examiner to identify the elemental compositions of the sample. This can be accomplished because there

are also discrete, characteristic quantities of energy released when the electron beam hits the sample. Each element has specific lines at specific energies that allow the examiner to determine the elemental composition of the sample.

Q: How does the automated software of computer-controlled SEM work?

A: Computer-controlled SEM allows the analysis of the surface of the entire sample in a relatively short amount of time. It reduces time and the human error associated with performing this type of examination manually. A manual analysis could take anywhere from 4 to 12 hours. The program analyzes the sample on a particle-by-particle basis and retains the individual feature characteristics and can relate the presence of Pb, Sb, or Ba to a single particle. When it detects particles with the presence of Pb, Sb, or Ba it flags the particles as potential GSR and stores images, compositions, and coordinate data. This enables the examiner to relocate to the particles of interest and confirm them manually after the automated analysis. The system generates a summary run sheet with stored image and elemental data to be used for relocation and confirmation applications.

Q: How is the computer-controlled SEM analysis calibrated?

A: The system is calibrated using known elemental standards.

Q: If unique or characteristic particles were detected in the submitted samples, can an examiner say that a particular individual fired this weapon?

A: One can never say a particular individual discharged a firearm, only that that individual was in an environment of primer residue.

Glossary

ACP Abbreviation for Automatic Colt Pistol, which describes the specific caliber in Colt self-loading pistols.

Action Operating mechanism of a particular firearm.

Ammunition The ammunition consists of a cartridge case, propellant, primer compound, and the projectile.

Annulus Ringlike space between the top of a primer and the case, created by the bevel of the primer pocket.

Antimony Metallic element with the chemical symbol Sb and atomic number 51. This element is commonly alloyed with lead to harden the bullet. It is also present as antimony sulfide in the primer mix.

Antimony Sulfide A common fuel of the primer compound used in cartridges.

Anvil Part of the primer that rests against the bottom of the primer pocket and against which the primer pellet is crushed when a firing pin strikes.

AP Abbreviation for armor piercing bullet.

Aperture The part of the beam that reaches the sample.

Assault Rifle Semi/full automatic, short-barreled military rifle.

Autoloading A gun that discharges, extracts, ejects, and reloads once with each action of the firing mechanism.

Automatic A firearm capable of ejecting a cartridge casing following discharge and reloading the next cartridge from the magazine.

Backscattered Electrons Produced by elastic collisions of incident electrons with atoms in the sample. The interaction with core electrons produces a backscattered signal that contains chemical information, reflected in the brightness of the particle reactive to the atomic number of elements, e.g., lead, atomic number 82, is brighter than iron, atomic number 26.

Ball Ammunition Military small arms ammunition with full-metal-jacket bullets; also known as “hard ball.”

Ballistics The science of the motion of projectiles, following the projectile travel from primer ignition to barrel exit, to target entry and until motion is stopped.

Barium Alkaline earth metal with chemical symbol Ba, atomic number 56. Present as barium nitrate in the primer.

Barium Nitrate A common oxidizer of the primer compound used in gun cartridges.

Barrel Discharge tube of a gun.

Battery cup Flanged metallic cup in a shotshell primer assembly providing support for the primer cup and anvil.

BB Air-rifle projectile of .177-in. diameter or a shotgun pellet of .18-in. diameter.

BBB Shot Shotgun pellet of .19-in. diameter.

Belted Case Cartridge case with an enlarged section that encircles the head, directly in front of the extractor groove.

Big Bore Any firearm or cartridge of .30 caliber or larger.

Black Powder Mixture of potassium nitrate, sulfur, and charcoal.

Blank A cartridge loaded with powder but not with a bullet.

Blow Back Leakage of gas rearward between the gun case and chamber wall, originating at the mouth of the case.

Body The part of a metallic cartridge case between the head and shoulder that contains the propellant.

Bolt Action A rifle action in which a manually operated sliding bolt acts as a breech block.

Bore The inside of a gun barrel.

Bore Diameter The diameter of the gun barrel before the rifling is cut.

Bottleneck Tapered shoulder of a gun case from the body to the neck, which gives the cartridge a bottle shape.

Breech The opening at the rear of the bore that permits a cartridge to be inserted.

Breechblock A movable block that closes the breech opening of a gun during firing.

Breech Face The part of the breech block that abuts the cartridge.

Buckshot Lead pellets ranging in size from .20-in. to .36-in. diameter.

Bullet A projectile fired from a firearm.

Caliber The diameter of either the bore or the projectile of a gun.

Cannelure Grooved section around the circumference of a bullet to allow crimping the case to the bullet.

Cap A now outdated term referring to the primer.

Cartridge A complete unit of assembled ammunition.

Cartridge Case In a firearm, a metal, plastic, or paper container that holds all the other components of a cartridge.

Chamber In a firearm, the part of the bore at the breech formed to accept a cartridge.

Choke The gradual decrease in bore diameter toward the muzzle of a shotgun barrel.

Cock The action of placing the firearm in the ready position by drawing back the hammer or bolt.

Condenser Lens Part of the scanning electron microscopy that demagnifies the electron beam and regulates its intensity.

Copper Metallic element with the chemical symbol Cu and atomic number 29 that commonly comprises “cartridge brass” which is typically 70% copper and 30% zinc (Zn).

Creep Movement of the bullet out of the cartridge case while in the cylinder or chamber.

Cylinder Rotating chambered breech of a revolver.

Depth of Field In microscopy, objects near and far remain in focus.

Dermal Nitrate Test *see* **Paraffin Test**

Detonate To explode with sudden violence.

Discharge To fire a gun.

Double Action When a single pull of the trigger both cocks and fires a weapon.

Double Base Powder A smokeless powder that contains both nitrocellulose and nitroglycerin.

EDS Abbreviation for Energy Dispersive Spectrometer, which detects X-rays for compositional analysis.

Ejection When a spent cartridge casing or unfired cartridge is automatically expelled from the breech of a firearm.

Ejection Port The opening in the side of a semi-automatic pistol that allows the cartridge case to be expelled from the gun.

Ejector A device in the breech loading mechanism which expels an empty casing or unfired cartridge from the breech.

Ejector Marks Markings left on the cartridge case from the action of the ejector.

Energy The amount of work done by a projectile at a given range, expressed in foot-pounds.

Express Cartridge A very large bore cartridge for heavy game, e.g., an elephant.

Extractor A device for pulling a spent or unfired cartridge from the chamber.

Extractor Groove The groove cut in front of the base of a rimless case to allow the case to be removed from the breech.

Extractor Mark Markings left on the cartridge case from the extractor.

Firing Pin The mechanism that strikes the primer to start ignition.

Flash Hole Vent leading from the primer pocket to the body of the cartridge case.

Flash Suppressor A device attached to the muzzle for the reduction of flash caused by the burning powder.

Full Automatic A weapon capable of continuous fire as long as the trigger is depressed.

Full Metal Jacket Bullet with the jacket completely enclosing the nose of the bullet.

Fulminate Original explosive mix used in primers as mercury fulminate. Generally abandoned with the introduction of brass and copper cartridge casings, because vaporized mercury attacked the brass, causing embrittlement.

Gauge A term used to identify the diameter of a smooth-bore (shotgun). It is defined the number of bore-diameter spherical lead balls needed to weigh 1 lb.

Gilding Metal A copper-zinc alloy used for jacketed bullets.

Grain Unit of measure defining the weight of a powder charge (437.5 grains to 1 oz. avoirdupois).

Grease Groove Circumferential groove on a lead bullet used to hold a lubricant.

Grips A pair of pieces designed to fit the frame of a weapon providing a form-fit gripping surface, usually plastic or wood.

Grooves Spiral cuts along the bore of a firearm that cause a projectile to spin as it travels through the barrel, providing stability in flight.

Gun In the SEM, the source of the electron beam.

Gunpowder Smokeless or black powder explosive propellant used in small arms.

Gunshot Residue Abbreviated as GSR, the particulate formed from the burning of the primer mix.

Hammer Pivotal metal part of firing mechanism designed to strike a firing pin or percussion cap to fire a weapon.

Handgun Pistol or revolver, firearm designed to carry in the hand.

Hand Loading Loading and reloading small-arms ammunition using hand-powered equipment.

Heel Rear portion of a bullet.

Ignition Time The time it takes the primer to set the propellant powder on fire.

Incendiary Bullet Containing a chemical agent designed to cause combustion.

Jacket Envelope enclosing the lead core of a bullet.

Land The spiraling raised portion of a bore that is formed when the grooves are fabricated.

Lead Element with the chemical symbol Pb and atomic number 82. Used in the fabrication of bullets and shot for its formability and lubrication properties.

Lead Styphnate A component in the primer mix that can be set off with a sharp blow, as when the firing pin strikes the primer cap.

Load A round of ammunition.

Long Rifle A .22 rimfire cartridge with a case length of .610 in. and an overall length of .990 in.

Luballoy A copper coating on lead handgun bullets to prevent barrel leading.

Magazine A container in a firearm that holds ammunition prior to chambering.

Magnum A large or high power cartridge.

Metal Fouling Deposition of bullet jacket material left in the barrel.

Misfire Ignition failure of a cartridge after primer has been struck by a firing pin.

Muzzle The end of the barrel of a firearm at a point where the projectile exits.

Muzzle Brake A device in a firearm used to reduce recoil and noise.

Muzzle Energy The energy level of a bullet as it exits the muzzle.

Muzzle Flash The burst of flame that is observed at the muzzle immediately after discharge.

Muzzle Loader A weapon in which the projectile and propellant are loaded directly into the muzzle of the firearm.

Nickel Nitrate Solution commonly used as a modifier in the atomic absorption analysis of gunshot residue.

Nitrocellulose A concentration of nitric and sulfuric acid on a cellulose material, e.g., cotton, used in the manufacture of smokeless powder.

Nitroglycerin A high explosive used in some propellant mixtures.

Objective Lens The part of a scanning electron microscope that focuses the electron beam onto the sample.

Obturation The act of sealing or preventing the escape of propellant gases from the breech of a gun.

Paraffin Test An early color test for the presence of nitrates also known as the “Dermal Nitrate Test.”

Pellet Another term for shot.

Percussion Cap A small metal cap with a priming mix that is placed on the nipple of a percussion lock.

Pistol A short-barreled firearm, designed for firing in one hand.

Powder A smokeless propellant of fine granulation discharged from a firearm.

Powder, Double Base *See Double Base Powder*

Powder, Semi-Smokeless An early powder consisting of nitrocellulose, potassium nitrate, charcoal, and sulfur.

Powder, Single Based A smokeless powder containing nitrocellulose.

Powder, Smokeless A powder containing nitrocellulose and perhaps other components, which does not produce a cloud of smoke when fired.

Primer A detonating mixture used to ignite a propellant charge.

Propellant An explosive charge for propelling a projectile.

Propellant Gases The gases created by the burning powder that force the projectile from the gun.

Range Horizontal travel of a projectile from weapon to the target; a location where firearms are used for testing the weapons integrity; skill of the shooter; practice or competitive events.

Recoil The backward movement or thrust of a gun caused by the pressure of the propellant gases in the process of pushing the projectile forward through the bore.

Reload The art of manually manufacturing ammunition.

Revolver A firearm containing a cylinder with chambers that revolve and fire sequentially.

Rifling The spiral grooves cut or formed through the bore of the barrel that put the spin on a projectile as it moves through the barrel.

Sabot A device, “shoe,” which enables a subcaliber projectile to be fired in a larger caliber barrel.

Safety A type of device that locks a weapon to prevent accidental discharge.

Saturation The point at which emission stops increasing as the filament drive is increased.

Scan Coils A device that deflects the electron beam to a rastering pattern over the sample.

Scanning Electron Microscope A microscope that utilizes an accelerated focused electron beam to image particulate samples (e.g., gunshot residue) at high magnification, with great depth-of-field, while providing size, shape, morphology, and chemical information in both manual and automated modes.

Secondary Electrons Inelastic interactions where electrons near the surface receive enough energy to exit the sample.

Semi-Automatic A firearm that has the capability of using force from an exploded round to eject a spent casing and to chamber the next round with an individual pull of the trigger required for each firing.

Shot Lead alloy pellets used as projectiles in shotguns.

Stigmator A device that corrects astigmatism defects in the optical system.

Striations Scoring or gouging streaks caused by movement of one object that is in contact with another object.

Terminal Velocity The speed of a projectile at the end of its flight, i.e., impact velocity.

Trace Metal Detection A factor related to the limits of detection of trace metals for a given analytical instrument.

Tracer Bullet A bullet that contains a pyrotechnic component ignited by the powder charge burn, leaving a visible trail of the flight path.

Trajectory The flight path of a projectile.

Trigger The mechanism used to release the firing pin of a firearm by applying pressure using a finger.

Trigger Guard A protective device consisting of a curved framework surrounding the trigger.

Velocity Speed of a projectile, usually measured in feet per second (fps).

Wad A paper or felt pad separating the charge from the projectile. Other carbonaceous materials are also used.

Wadcutter A specially designed bullet, shaped to cut a clean hole in target paper.

Bibliography

- Aginsky, V. and Sorokina, G., Time of shooting — feasibility of discriminating “fresh” and “old” organic gunshot residues, Abstract of paper presented at the 14th Meet. of the Int. Assoc. of Forensic Sci. (IAFS), Tokyo, Japan, Aug. 1996.
- Albu-Yaron, A. and Amiel, S., *J. Radioanal. Chem.*, 11(1), 123–132, 1972.
- Andersen, C. A. and Hasler, M. F., X-ray optics and microanalysis, 4th Int. Cong. on X-ray Optics and Microanalysis, Hermann, Paris, 1966, 310.
- Anderson, N. D. and Brown, W. R., *Fireworks!* Dodd, Mead, and Co., New York, 1983.
- Andrasko, J., Characterization of smokeless powder flakes from fired cartridge case and from discharge patterns on clothing, *J. Forensic Sci.*, 37, 1030–1047, 1992.
- Andrasko, J. and Maehly, A. C., Detection of gunshot residues on hands by scanning electron microscopy, *J. Forensic Sci.*, 22, 279–287, 1977.
- Andrasko, J. and Pettersson, S., A simple method for collection of gunshot residues from clothing, *J. Forensic Sci.*, 31, 321–330, 1991.
- Andrasko, J., Norberg, T., and Stahling, S., Time since discharge of shotguns. *J. Forensic Sci.*, 43(5), 1005–1015, 1998.
- Annual Book of ASTM Standards, Vol. 3.01.
- Annual Book of ASTM Standards, Vol. 3.06.
- Annual Book of ASTM Standards, Vol. 14.02.
- Archer, A.W., Separation and identification of minor components in smokeless powders by thin-layer chromatography, *J. Chromatogr.*, 108, 401–404, 1975.
- Arnold, R., *The Book of the .22*, Nicholas Kaye Limited, London, U.K., 1962.
- Arthur, C., Killam, L., Buchholz, K., Pawliszyn J., and Burg, J. R., Automation and optimization of solid-phase microextraction, *Anal. Chem.*, 64(17), 1960–1966, 1992.
- Ashrof-Khorassni, M. and Taylor, L. T., Qualitative supercritical fluid chromatography/ Fourier transform infrared spectroscopy study of methylene chloride and supercritical carbon dioxide extracts of double-base propellant, *Anal. Chem.*, 61, 145–148, 1989.
- Association of Industrial Explosives, Ed., *The Handbook of Explosives*, Kyoritsu Press, Tokyo, 1987.
- ASTM Designation: E 1508–93a. Standard guide for quantitative analysis by energy-dispersive spectroscopy, 919–925, Oct. 1993.

- ASTM Designation: E 1588–95. Standard guide for gunshot residue analysis by scanning electron microscopy/energy-dispersive spectroscopy, 1006–1008, May 1995.
- Barbour, R. T., *Pyrotechnics in Industry*, McGraw Hill, New York, 1982, 23–25.
- Barnes, R.W., Identification of the nitrate esters of five alkyl alcohol by thin-layer chromatography, *J. Chromatogr.*, 31, 606–608, 1967.
- Bashinski, J. S., The evaluation of gunshot residues, the sodium rhodizonate test, Presented at the CA Assoc. of Criminalists Fall Seminar, Berkeley, CA, Oct. 1974.
- Bashinski, J. S., Davis, J. E., and Young, C., Detection of lead in gunshot residues on targets using the sodium rhodizonate test, *AFTE J.*, 6(4), 5–6, 1974.
- Basu, S. and Ferriss S., A refined collection technique for rapid search of gunshot residue particles in the SEM, *Scanning Electron Microsc.*, 1, 375–384, 392, 1980.
- Basu, S., Formation of gunshot residues, *J. Forensic Sci.*, 27(1), 72–91, 1982.
- Basu, S., Fundamental studies of gunshot residues by “Glue-Lift,” Proceedings of Amer. Acad. of Forensic Sci., 35th Annu. Meet., Cincinnati, OH., 49, Feb. 15–19, 1983.
- Basu, S., The mechanism of gunshot residue deposition and its probing characteristics to reconstruct shootings, Bailey, G. W., Ed., Proceedings of the 43rd Annu. Meet. of the Electron Microsc. Soc. of Am., Louisville, KY, Aug. 5–9, 1985, San Francisco Press, Inc., San Francisco, CA, 104–107, 1985.
- Basu, S., The mechanism of gunshot residue deposition and its probing characteristics to reconstruct shootings, Basu, S. and Millette, J. R., Eds. *Electron microscopy in Forensic, Occupational, and Environmental Health Sciences*, Plenum Press, New York, 1986, 1–22.
- Basu, S., Ferriss, S., and Horn, R., Suicide reconstruction by glue-lift of gunshot residue, *J. Forensic Sci.*, 29, 843–864, 1984.
- Basu, S., Boone, Jr., C. E., Denio, Jr., D. J., and Miazga, R. A., Fundamental studies of gunshot residue deposition by glue-lift, *J. Forensic Sci.*, 42(4), 571–581, 1997.
- Beijer, R., Experiences with Zincon, a useful reagent for the determination of firing range with respect to leadfree ammunition, *J. Forensic Sci.*, 39, 981–987, 1994.
- Bellanca, S., Buchanan, J. D., Guinn, V. P., Magliore, J. C., and Ruch, R. R., Applications of neutron activation analysis in scientific crime detection, Rep. No. GA-3491, Gulf General Atomic, Inc., U.S. Atomic Energy Commission, San Diego, CA, Apr. 1962.
- Berberich, D. W., Yost, R. A., and Fetterolf, D., Analysis of explosives by liquid chromatography/thermospray/mass spectrometry, *J. Forensic Sci.*, 33, 946–959, 1988.
- Bergman, P., Enzel, P., and Springer, E., The detection of gunshot residue on the bottom of discharged bullets, *J. Forensic Sci.*, 33(4), 960–968, 1988.
- Bhattacharyya, C. N., Dispersion of firing discharge residues using a Maxwellian model, *Forensic Sci. Int.*, 42, 271–277, 1989.
- Boehm, E., Application of the SEM in forensic medicine, Proceedings of the 4th Annu. Scan. Electro. Microsc. Symp., O. Johari and I. Corvin, Eds., Ill. Instit. of Technol. Res. Instit., Chicago, IL, 1971, 553–560.

- Bonfanti, M. S. and De Kinder, J., The influence of manufacturing processes on the identification of bullets and cartridge cases — a review of the literature, *Sci. Justice*, 39(1), 3–10, 1999.
- Booker, J. I., A method for the identification of smokeless powders and their residues by thin-layer chromatography of their minor constituents, *J. Forensic Sci. Soc.*, 13, 199–201, 1973.
- Booker, J. L. and Schroeder, D. D., A note on the variability of barium and antimony levels in cartridge primers and its implication for gunshot residue identification, *J. Forensic Sci. Soc.*, 24(2), 81–84, 1984.
- Bradley, J. D., Mining and milling methods and costs at the Yellow Pine Mine, Stibnite, Idaho. BuMines Inf. Circ. 7194, 12, 1942.
- Bratin, K., Kissinger, P. T., Briner, R. C., and Bruntlett, G. S., Determination of nitro aromatic, nitramine and nitrate ester explosive compounds in explosive mixtures and gunshot residue by liquid chromatography and reductive electrochemical detection, *Anal. Chim. Acta*, 130, 295–311, 1981.
- Bray, J. L., *Nonferrous Production Metallurgy*, 2nd ed., John Wiley & Sons, New York, 1959, 72–82.
- Brihaye, C. I., Machiroux, R., and Gillain, G., Gunpowder residues detection by anodic stripping voltammetry, *Forensic Sci. Int.*, 20, 269–276, 1982.
- Brock, *A History of Fireworks*, ASTH, George G. Harrap and Co., 1949, 137–164.
- Brown, H., Cauchi, D. M., Holden, J. L., Allen, F. C., Cordner, S., and Thatcher, P., Image analysis of gunshot residue in entry wounds. II — A statistical estimation of firing range, *Forensic Sci. Int.*, 100(3), 179–186, 1999.
- Brown, H., Cauchi, D. M., Holden, J. L., Allen, F. C., Cordner, S., and Thatcher, P., Image analysis of gunshot residue on entry wounds. I — The technique and preliminary study, *Forensic Sci. Int.*, 100(3), 163–177, 1999.
- Brown, H., Cauchi, D. M., Holden, J. L., Wrobel, H. A., and Cordner, S., Image analysis of gunshot residue on entry wounds. I — the technique and preliminary study, *Forensic Sci. Int.*, in press.
- Bydal, B. A., Percussion primer mixes, *Assoc. Firearms Toolmark Examiners J.*, 22, 1–26, 1990.
- Camscan, *Scan. Electron Microsc. – Operating and Technical Information Manual*, Series 4, 1983.
- Capannesi, G., Ciavola, C., and Sedda, A. F., Determination of firing distance and firing angle by neutron activation analysis in a case involving gunshot wounds, *Forensic Sci. Int.*, 61, 75–84, 1993.
- Casuccio, G. S., Schwoeble, A. J., Henderson, B. C., Lee, R. J., Hopke, P. K., and Sverdrup, G. M., The use of CCSEM and microimaging to study source/receptor relationships, APCA Receptor Models in Air Resources Management Specialty Conference, San Francisco, CA, Feb. 24–26, 1988.
- Charpentier, B. and Desrochers, C., Analysis of primer residue from lead free ammunition by X-ray microfluorescence, *J. Forens. Sci.*, 45(2), 447–452, 2000.

- Cheng, W. W. and Lee, R. J., Step shape \emptyset (PZ) model and on-standard ultra light element analysis program, 1988 Joint Meet. of the Electron Microsc. Soc. of Amer., Microbeam Anal. Soc., Microscop. Soc. of Canada, Milwaukee, WI, Aug. 7–12, 1988.
- Cole, M. D., Ross, N., and Thorpe, J. W., Gunshot residue and bullet wipe detection using a single lift technique, *AFTE J.*, 24, 254, 1992.
- Collins, D. A., Modification to a thermal energy analyzer with associated electronic filtering for improved gas chromatographic analysis of explosives traces, *J. Chromatogr.*, 483, 379–383, 1989.
- Cone, R. D., Detection of barium, antimony and lead in gunshot residue, *Police Weapons Center Bull.*, 4, 1973.
- Conkling, J. A., *Chemistry of Pyrotechnics*, Marcel Dekker, New York, 1985, 143–165.
- Conkling, J. A., Pyrotechnics, *Sci. Am.*, 96–102, July 1990.
- Cooper, R., Guileyardo, J. M., Stone, I. C., Hall, V., and Fletcher, L., Primer residues deposited by handguns, *Am. J. Forensic Med. Pathol.*, 15, 325–327, 1994.
- Cornelis, R. and Timperman, J., Gun firing detection method based on Sb, Ba, Pb, and Hg deposits on hands: evaluation of credibility of the test, *Med. Sci. Law*, 14(2), 98–116, 1974.
- Court of Appeal, State of California, First Appellate Dist., Div. 4, Case No. 1, Crim. 16437 (Superior Ct. No. 62941). *People v. Palmer*: investigating criminalist, W. Fong of the Santa Clara County Crime Lab.
- Cowan, M. E. and Purdon, P. L., A study of the paraffin test, *J. Forensic Sci.*, 12(1), 19–36, 1967.
- Cowan, M. E., Purdon, P. L., Hoffman, C. M., Brunelle, R., Gerber, S. R., et al., *J. Radioanal. Chem.*, 15(1), 203–218, 1973.
- Crow, G. A., Christman, L., and Utlaut, M., A focused ion beam secondary ion mass spectroscopy system, *J. Vac. Sci. Technol. B*, 13(6), 2607–2612, 1995.
- Dahl, D. B. and Lott, P. F., Determination of black and smokeless powder residues in firearms and improved devices, *Microchem. J.*, 35, 40–50, 1987.
- Dahl, D. B. and Lott, P. F., Gunshot residue determination by means of gunpowder stabilizers using high performance liquid chromatography with electrochemical detection and analysis of metallic residues by graphite furnace atomic absorption spectrophotometry, *Microchem J.*, 35, 347–359, 1987.
- Dahl, D. B., Clayton, J. C., and Lot, P. F., Gunshot residue analysis – an applicability study, *Microchem. J.*, 35, 360–364, 1987.
- de Kok, A., Roorda, I. M., Frei, R. W., and Brinkman, U. A. T., Gas chromatographic analysis of phenylurea herbicides following catalytic hydrolysis on silica gel, *Chromatographia*, 14, 579–586, 1981.
- de Kok, A., Vos, Y. J., van Gardoren, C., de Hong, T., van Opstal, M., and Frei, R. W., et al., Chromatographic determination of phenylurea herbicides and their corresponding aniline degradation products in environmental samples, I. *J. Chromatogr.*, 288, 71–89, 1984.

- DeGaetano, D. and Siegel, J. A., Survey of gunshot residue analysis in forensic science laboratories, *J. Forensic Sci.*, 35(5), 1087–1095, 1990.
- DeGaetano, D., Siegel, J. A., and Klomparens, K. L., A comparison of three techniques developed for sampling and analysis of gunshot residue by scanning electron microscopy/energy dispersive X-ray analysis (SEM EDX), *J. Forensic Sci.* 37(1), 281–300, 1992.
- Deinet, W. and Lezczynski, C. H., Examinations to determine close-range firing distances using a process control computer, *Forensic Sci. Int.*, 31, 41–54, 1986.
- Diederichs, R., Camp, M. J., Wilimovsky, A. E., Haas, M. A., and Dragen, F. R., Investigations into the adaptability of scanning electron microscopy and X-ray fluorescence spectroscopy to firearms related examinations, *Assoc. Firearms Toolmarks Examiners J.*, 6, 1974.
- Douse, J. M. F., Trace analysis of explosives in handswab extracts using amberlite XAD-7 porous polymer beads, silica capillary column gas chromatography with electron-capture detection and thin-layer chromatography, *J. Chromatogr.*, 234, 415, 1982.
- Douse, J. M. F., Trace analysis of explosives at low picogram level using silica capillary column gas chromatography with thermal energy analyzer detection, *J. Chromatogr.*, 256, 359–362, 1983.
- Douse, J. M. F., Trace analysis of explosives at low nanogram level in handswab extracts using columns of Amberlite XAD-7 porous polymer beads with silica capillary column gas chromatography with thermal energy analysis and electron-capture detection, *J. Chromatogr.*, 328, 155–165, 1985.
- Douse, J. M. F., Improved method for the trace analysis of explosives by silica capillary column gas chromatography with thermal energy analysis detection, *J. Chromatogr.*, 410, 181–189, 1987.
- Douse, J. M. F., Trace analysis of explosives by capillary supercritical fluid chromatography with thermal energy analysis detection, *J. Chromatogr.*, 445, 244–250, 1988.
- Douse, J. M. F., Dynamic headspace method for the improved clean-up of gunshot residues prior to the detection of nitroglycerine by capillary column gas chromatography with thermal energy analysis detection, *J. Chromatogr.*, 464, 178–185, 1989.
- Douse, J. M. F. and Smith, R. N., Trace analysis of explosives and firearm discharge residues in the Metropolitan Police Forensic Science Laboratory, *J. Energetic Mater.*, 4, 169–186, 1986.
- Eisele, J. W., Reay, D. T., and Cook, A., Sites of suicidal gunshot wounds, *J. Forensic Sci.*, 26(3), 480–485, 1981.
- Engelhardt, H., Zapp, J., and Kolla, P., Sample preparation by supercritical fluid extraction in environmental food and polymer analysis, *Chromatographia*, 32, 527–537, 1991.
- Engelhardt, H., Meister, J., and Kolla, P., Optimization of post-column reaction detector for HPLC of explosives, *Chromatographia*, 35, 5–12, 1993.

- Espinoza, E. O'N and Thornton, J. I., Characterization of smokeless powder by means of diphenylamine stabilizer and its nitrated derivatives, *Anal. Chim. Acta*, 288, 57–69, 1994.
- Fields, E. A., A survey of the advantages and disadvantages of different types of specimen mounts used to sample gunshot residue, Presented at the IAFS meeting, 1999.
- Fine, D. H., Lieb, D., and Rufeh, F., Principle of operation of the thermal energy analyzer for the trace analysis of volatile and non-volatile nitroso compounds, *J. Chromatogr.*, 107, 351–357, 1975.
- Fine, D. H., Yu, W. C., Goff, E. U., Bender, E. C., and Reutter, D. J., Picogram analyses of explosives residues using the thermal energy analyzer (TEA), *J. Forensic Sci.*, 29, 732–746, 1984.
- Fiori, C. E., Newbury, D. E., and Myklebust, R. L., Artifacts observed in energy dispersive X-ray spectrometry in electron beam instruments — a cautionary guide. NIST Special Publication 604, *Proceedings of the Workshop on Energy Dispersive Spectrometry*, National Institute of Standards and Technology, Gaithersburg, MD 1981.
- Fireworks, Dialogue Systems Inc. in association with A&E, George Pimpton — video narrator 1994.
- Fischbeck, H. J., Ryan, S. R., and Snow, C. C., Detection of bullet residue in bone using proton-induced X-ray emission (PIXE) analysis, *J. Forensic Sci.*, 31(1), 79–85, 1986.
- Fisher, B. A. J., *Techniques of Crime Scene Investigation*, 5th ed., Elsevier, New York, 1992, 271–303, 305–328.
- Frei, R. W., Lawrence, J. F., and Legay, D. S., The analysis of carbamate and urea herbicides by fluorometry of their dansylated amine moieties, *Analyst*, 98, 9–18, 1973.
- Frost, G. E., Ammunition making – An insider's story, National Rifle Association, Washington, D.C., 47–69, May 1990.
- Gansau, H. and Becker, U., Semi automatic detection of gunshot residues by scanning electron microscopy and energy dispersive X-ray analysis (SEM EDX), *Scanning Electron Microsc.*, 1, 107–114, 179–184, 1982.
- Garofano, L., Capra, M., Ferrari, F., Bizzaro, G. P., Tullio, D. D., Dell'Olio, M., and Ghitti, A., Gunshot residue, further studies on particles of environmental and occupational origin, *Forensic Sci. Int.*, 103(1), 1–21, 1999.
- Germani, M. S., Evaluation of instrumental parameters for automated scanning electron microscopy gunshot residue particle analysis, *J. Forensic Sci.*, 36, 331–342, 1991.
- Giacalone, J. R., Particle analysis of gunshot residue via SEM EDS with the use of carbon-conductive tapes, presented at NWAFFS, May 1995.
- Giacalone, J. R., Scanning electron microscopy/energy dispersive X-ray analysis (SEM EDS) of forensic microtrace particles collected on carbon-conductive adhesive tape, *Scanning*, 19(3), 232–233, 1997.

- Giacalone, J. R., Forensic fireworks analysis and their residue by scanning electron microscopy/energy-dispersive spectroscopy, *Scanning*, 20(3), 172–173, 1998.
- Giacalone, J. R., Forensic particle analysis of microtrace pyrotechnic residue, *Scanning*, 21(2), 100–101, 1999.
- Gislason, J. and Pate, B. D., Studies of gunshot residue, *J. Radioanal. Chem.*, 15(1), 103–113, 1983.
- Goad, K. J. W. and Halsey, D. H. J., *Ammunition (Including Grenades and Mines)*, Brassey's Publishers, Oxford, U.K., 1982, 49.
- Goldstein, J. I., Newbury, D. E., Eclin, P., Joy, D. C., Romig, Jr., A. D., Lyman, C. D., Fiori, C., and Lifshin, E., *Scanning Electron Microscopy and X-ray Microanalysis*, 2nd ed., Plenum Press, New York, 1992.
- Goleb, J. A. and Midkiff, Jr., C. R., Firearms discharge residue sample collection techniques, *J. Forensic Sci.*, 20(4), 701–707, 1975.
- Goleb, J. A., and Midkiff, Jr., C. R., The determination of barium and antimony in gunshot residue by flameless atomic absorption spectroscopy using a tantalum strip atomizer, *Appl. Spectrosc.*, 29(1), 44–48, Jan.-Feb. 1975.
- Griest, W. H., Guzman, C., and Dekker, M., Packed-column supercritical fluid chromatographic separation of highly explosive compounds, *J. Chromatogr.*, 467, 423–429, 1989.
- Grob, K. and Schilling, B., Uncoated capillary column inlets (retention gaps) in gas chromatography, *J. Chromatogr.*, 391, 3–18, 1987.
- Grove, C. A., Judd, G., and Horn, R., Examination of firing pin impressions by scanning electron microscopy, *J. Forensic Sci.*, 17(4), 645–58, 1972.
- Grove, C. A., Judd, G., and Horn, R., Evaluation of SEM potential in the examination of shotgun and rifle firing pin impressions, *J. Forensic Sci.*, 19(3), 441–447, 1974.
- Guileyardo, J. M., Stone, I. C., and Odom, C. B., Gunshot residue, ten years later, *Am. J. Forensic Med. Pathol.*, 13(1), 88, 1992.
- Guinn, V. P., Applications of nuclear science in crime investigation, *Annu. Rev. Nucl. Sci.*, 24, 561–591, 1974.
- Guinn, V. P., Recent studies involving bullet-lead fragments, Presented at the 49th Semi-annual Seminar, CA Assoc. of Criminalists. Indian Wells, CA, May 12-14, 1977.
- Guinn, V. P., Lukens, H. R., and Schlesinger, H. L., Application of NAA in scientific crime investigations, Rep. GA-9807, Gulf General Atomic, Inc., San Diego, CA, 65.
- Gunaratnam, L. and Himberg, K., The identification of gunshot residue particles from lead-free Sintox ammunition, *J. Forensic Sci.*, 39, 532–536, 1994.
- Halberstam, R. C., A simplified probability equation for gunshot primer residue (GSR) detection, *J. Forensic Sci.*, 36, 894–897, 1991.
- Harris, A., Analysis of primer residue from CCI Blazer lead free ammunition by scanning electron microscopy/energy-dispersive X-ray, *J. Forensic Sci.*, 40, 27–30, 1995.

- Harrison, H. C. and Gilroy, R., Firearms discharge residues, *J. Forensic Sci.*, 4(2),184–199, 1959.
- Hatcher, J. S., Jury, F., and Weller J., *Firearms Investigation, Identification and Evidence*. T. Samsworth, Ed., The Stackpole Company, Harrisburg, PA, 1957.
- Havakost, D. G., Peters, C. A., and Koons, R. D., Barium and antimony distributions on the hands of nonshooters, *J. Forensic Sci.*, 35(5), 1096–1114, 1990.
- Heard, B. J., *Handbook of Firearms and Ballistics — Examining and Interpreting Forensic Evidence*, John Wiley & Sons, New York, 1997, 62–68.
- Hellmiss, G., Lichtenberg, W., and Weiss, M., Investigation of gunshot residues by means of Auger electron spectroscopy, *J. Forensic Sci.*, 32, 747–760, 1987.
- Hoffman, C. M. Neutron activation analysis for the detection of firearm discharge residue collected on cotton swabs, *J. Assoc. Off. Anal. Chem.*, 56, 1388, 1975.
- Hoffman, C. M. and Wilder, R. L., *Am. Rifleman*, 119, 26–27, 1971.
- Hoffman, R., Use of the film impression procedure – a procedure for the detection of surface distribution of chemical elements in gunshot residue, *Arch Kriminol.*, 175(1–2), 21–30, 1985.
- Hoover, M. R., White, E. W., Lebiezick, J., and Johnson, Jr. G. C., Automated characterization of particulates and inclusions by computer-controlled SEM probe, *Proc. of the 10th Annu. Microbeam Anal. Soc. Conf.*, 1975.
- Hudlicky, M., *Reductions in Organic Chemistry*, Ellis Horwood Limited, London, U.K., 1984, 71.
- Hudson, P., Suicide with two guns fired simultaneously, *J. Forensic Sci.*, 27(1), 6–7, 1982.
- Hyma, B. A., Gunshot residue and range of fire, *Am. J. Forensic Med. Pathol.*, 13(2), 175–177, 1992.
- Jalanti, T., Henchoz, P., Gallusser, A., and Bonfanti, M. S., The persistence of gunshot residue on shooters' hands, *Sci. Justice*, 39(1), 48–52, 1999.
- Jane, I., Brooks, P. G., Douse, J. M. F., and O'Callaghan, Detection of gunshot residues via analysis of their organic constituents, *Proc. of the Int. Symp. on the Anal. and Detection of Explosives*, Quantico, VA, 1983, 475.
- Johari, O. and DeNee, P. B., Handling, mounting and examination of particles for scanning electron microscopy, *Scanning Electron Microsc.*, 1, 250–256, 1972.
- Johnson, D. L., McIntyre, B. L., Fortmann, R., Stevens, R. K., and Hanna, R. B., A chemical element comparison of individual particle analysis and bulk analysis methods, *Scanning Electron Microsc.*, 1, 469, 1981.
- Johnson, Jr., G. G. and White, E. W., X-ray emission wavelengths and KeV tables for nondiffractive analysis, *ASTM Data Series DS45*, ASTM, Philadelphia, PA, 1970.
- Jones, P. F. and Nesbitt, R. S., A photoluminescence technique for the detection of gunshot residue, *J. Forensic Sci.*, 20(2), 231–241, 1975.
- Jones, P. F., Nesbitt, R. S., and Wessel, J. E., Characterization of gunshot residue by particle analysis techniques, The Aerospace Corporation, USA, El Segundo, CA, 1974.

- Judd, G., Sabo, J., Hamilton, W., Ferriss, S., Horn, R., SEM microstriation characterization of bullets and contaminant particle identification, *J. Forensic Sci.*, 19(4), 789–811, 1984.
- Karger, B., Differentiation of hunting accident and suicide with rifles, *Versicherungsmedizin*, 48(1), 11–15, 1996.
- Kee, T. G. and Beck, C., Casework assessment of an automated scanning electron microscope/microanalysis system for the detection of firearm discharge particles. *J. Forensic Sci. Soc.*, 27(5), 321–330, 1987.
- Kee, T. G., Halmes, D. M., Doolan, K., Hamill, J. A., and Griffin, R. M. E., The identification of individual propellant particles, *J. Forensic Sci. Soc.*, 30, 285–292, 1990.
- Keeley, R. H., Some applications of electron probe instruments in forensic science, *Proc. of the Anal. Div. of the Chem. Soc.*, 13, 178–181, June 1976.
- Keeley, R. H. and Nolan, P. J., *Automatic Particle Analysis*, Metropolitan Police Lab. Rep. and Rep. to Interpol Meet., Paris, London Metropolitan Police. London, U.K., 1986.
- Keisch, B. and Callahan, R. C., Potential uses of lead isotope ratios in gunshot cases, *J. Assoc. Anal. Chem.*, 61(3), 520–525, 1978.
- Keto, R. O., Comparison of smokeless powders by pyrolysis capillary gas chromatography and pattern recognition, *J. Forensic Sci.*, 34, 74–82, 1989.
- Kilty, J. W., Activity after shootings and its effect on the retention of primer residue, *J. Forensic Sci.*, 20, 219–230, 1975.
- Kilty, J. W., A review of the FBI laboratory's gunshot primer residue program, *Crime Lab. Dig.*, 13(2), 54–61, Apr. 1986.
- Kilty, J. W. and Matricardi, V. R., Gunshot residue particles detected vs. quantity of primer residue present, Pres. at the 28th Annu. Meet. of the Amer. Acad. of Forensic Sci., Washington, D.C., Feb. 1976.
- Kinard, W. D. and Midkiff, C. R., Jr., The application of oxygen plasma ashing to gunshot residue analysis, *J. Forensic Sci.*, 23(2), 288–303, 1977.
- King, R. M., The work of the explosives & gunshot residues unit of the forensic science service (UK). Yinon, J., Ed., *Advances in Analysis and Detection of Explosives*, Kluwer Academic Publishers, Dordrecht, the Netherlands, 1993, 91–100.
- Klingenberg, G., Gun muzzle research at Fraunhofer Institute EMI-AFB, *J. Ballistics*, 9, 2129–2167, 1986.
- Kohlbeck, J. A., Determination of nitroglycerin and resorcinol in double-base propellant following separation by thin-layer chromatography, *Anal. Chem.*, 37, 1282–1283, 1965.
- Koons, R. D., ICP atomic spectrometry in the forensic laboratory, *Spectroscopy*, 8(6), 16–21, 1993.
- Koons, R. D., Flameless atomic absorption spectrophotometric determination of antimony and barium in gunshot residue collection swabs — a collaborative study, *Crime Lab. Dig.*, 20, 19–23, 1993.

- Koons, R. D., Analysis of gunshot primer residue collection swabs by inductively couple plasma-mass spectrometry, *J. Forensic Sci.*, 43(4), 748–754, 1998.
- Koons, R. D., Havekost, D. G., and Peters, C. A., Analysis of gunshot primer residue collection swabs using flameless atomic absorption spectrophotometry — a re-examination of extraction and instrument procedures, *J. Forensic Sci.*, 32(4), 846–865, 1987.
- Koons, R. D., Havekost, D. G., and Peters, C. A., Determination of barium in gunshot residue collection swabs using inductively coupled plasma-atomic emission spectrometry, *J. Forensic Sci.*, 33(1), 35–41, 1988.
- Koons, R. D., Havekost, D. G., and Peters, C. A., Analysis of gunshot primer residue collection swabs using flameless atomic absorption spectrophotometry and inductively coupled plasma — atomic emission spectrometry — effects of a modified extraction procedure and storage of standards, *J. Forensic Sci.*, 34(1), 218–221, 1989.
- Krishnan, S. S., Determination of gunshot firing distances and identification of bullet holes by neutron activation analysis, *J. Forensic Sci.*, 12, 112–122, 471–483, 1967.
- Krishnan, S. S., Detection of gunshot residues on the hands by activation and atomic absorption analysis, *J. Forensic Sci.*, 19, 351–356, 780–797, 1974.
- Krishnan, S. S., Detection of gunshot residue on the hand by trace element analysis, *J. Forensic Sci.*, 22, 304–324, 1977.
- Krishnan, S. S., Detection of gunshot residue: present status, Saferstein, R., Ed., *Forensic Science Handbook*, Prentice-Hall, Englewood Cliffs, NJ, 1982, 1, 572–591.
- Krishnan, S. S., Trace element analysis by atomic absorption spectrometry and neutron activation analysis in the investigation of shooting cases, *J. Can. Soc. Forensic Sci.*, 6(2), 55–77, 1983.
- Krishnan, S. S., Gillespie, K. A., and Anderson, E. J., Rapid detection of firearm discharge residues by atomic absorption and neutron activation analysis, *J. Forensic Sci.*, 16, 144–151, 1971.
- Kumarawickrama, R. A. D., The characterization and identification of organic gunshot residue, M.Sc. thesis, University of Strathclyde, U.K., 1985.
- Lafleur, A. L. and Mills, K. M., Trace level determination of selected nitro-aromatic compounds by gas chromatography with pyrolysis/chemiluminescent detection, *Anal. Chem.*, 53, 1202–1205, 1981.
- Lantos, J., Brinkman, U. A. T., and Frei, R. W., Residue analysis of methoxuron and its breakdown product (3-chloro-4-methoxyaniline) by thin-layer and high-performance liquid chromatography with fluorescence detection, *J. Chromatogr.*, 292, 117–127, 1984.
- Lantz, P. E., Jerome, W. G., and Jaworski, J. A., Radiopaque deposits surrounding a contact small-caliber gunshot wound, *Am. J. Forensic Med. Pathol.*, 15(1), 10-3, 1994.
- Lawrence, J. F. and Laver, G. W., Analysis of carbamate and urea herbicides in foods, using fluorogenic labeling, *J. Assoc. Off. Anal. Chem.*, 57, 1022–1025, 1974.

- Lebiedzki, J. and Johnson, D. L., Rapid search and quantitative analysis of gunshot residue particles in the SEM, *J. Forensic Sci.*, 45(1), 83–92, 2000.
- Lee, R. J. and Fisher, R. M., Quantitative characterization of particulates by scanning and high voltage electron microscopy, *National Bur. of Stand. Spec. Publ.*, 533, 1980.
- Lee, R. J. and Kelly, J. F., Back-scattered electron imaging for automated particle analysis, *Microbeam Analysis*, D. E. Newbury, Ed., San Francisco Press, San Francisco, CA, 1979.
- Lee, R. J. and Kelly, J. F., Applications of SEM-based automated image analysis, *Microbeam Analysis*, D. B. Wittry, Ed., San Francisco Press, San Francisco, CA, 1980.
- Lee, R. J. and Kelly, J. F., Overview of SEM-based automated image analysis, *Scanning Electron Microsc.*, 1, 303, 1980.
- Lee, R. J., DeBray, D. R., and Lentz, S., The SEM in industry: applications and limitations, Presented at the Conf. on Mater. Characterization: Pract. Appl., sponsored by ASM, Oak Brook, IL, Sept.17-19, 1975.
- Lee, R. J., Spitzig, W. A., Kelly, J. F., and Fisher, R. M., Quantitative metallography by computer-controlled scanning electron microscopy, *J. Metals*, 33(3), 80, 1981.
- Leggett, L. S. and Lott, P. F., Gunshot residue analysis via organic stabilizers and nitrocellulose, *Microchem. J.*, 39, 76–85, 1989.
- Li, K. C., Arsenic and antimony: modern uses of nonferrous metals, *Am. Inst. Min. Met. Eng.*, 2nd ed., 40–53, 1953.
- Liao, Z. and Wu, J., Gunshot residue particle detected in entrance wound of near range fire with energy dispersive X-ray, *Fa I Hsueh Tsa Chih*, 13(3), 152–153, 1997.
- Lichtenberg, W., Determination of gunshot residues (GSR) in biological samples by means of Zeeman atomic absorption spectroscopy, *Fresenius Z. Anal. Chem.*, Ger. 328, 367–369, 1987.
- Lichtenberg, W., Methods for the determination of shooting distance, *Forensic Sci. Rev.*, 2, 37–62, 1990.
- Liu, J. H., Lin, W. F., and Nicol, J. D., The application of anodic stripping voltammetry to forensic science: II anodic stripping voltammetric analysis of gunshot residues. *Forensic Sci. Int.*, 16, 53–62, 1980.
- Lloyd, J. B. F., Clean-up procedures for the examination of swabs for explosives traces by high-performance liquid chromatography with electrochemical detection at a pendant mercury drop electrode, *J. Chromatogr.*, 261, 391–406, 1983.
- Lloyd, J. B. F., High-performance liquid chromatography of organic explosives components with electrochemical detection at a pendant mercury drop electrode, *J. Chromatogr.*, 257, 227–236, 1983.
- Lloyd, J. B. F., Detection and differentiation of nitrocellulose traces of forensic science interest with reductive mode electrochemical detection at a pendant mercury drop electrode coupled with size-exclusion chromatography, *Anal. Chem.*, 56, 1907–1912, 1984.

- Lloyd, J. B. F., Microcolumn clean-up and recovery techniques for organic explosives compounds and for propellants traces in firearms discharge residues, *J. Chromatogr.*, 330, 121–129, 1985.
- Lloyd, J. B. F., Adsorption characteristics of organic explosives compounds on adsorbents typically used in clean-up and related trace analysis techniques, *J. Chromatogr.*, 328, 145–154, 1985.
- Lloyd, J. B. F., Adsorption and exclusion characteristic of nitrocellulose with reference to microcolumn clean-up technique for the detection of propellants traces in firearms discharge residues, *J. Chromatogr.*, 351, 323–330, 1986.
- Lloyd, J. B. F., Glyceryl dinitrates in the detection of skin contact with explosives and related materials of forensic science interest, *J. Forensic Sci. Soc.*, 26, 341–348, 1986.
- Lloyd, J. B. F., Liquid chromatography of firearms propellants traces, *J. Energetic Mater.*, 4, 239–271, 1986.
- Lloyd, J. B. F., Liquid chromatography with electrochemical detection of explosives and firearms propellant traces, *Anal. Proc.*, 24, 239–240, 1987.
- Lloyd, J. B. F., Forensic explosives and firearms traces: Trapping HPLC peaks for gas chromatography, *J. Energetic Mater.*, 9, 1–17, 1991.
- Lloyd, J. B. F. and King, R. M., One-pot processing of swabs for organic explosives and firearms residue traces, *J. Forensic Sci.*, 35, 956–959, 1990.
- Loper, G. L., Calloway, A. R., Stamps, M. A, Wolten, G. M, and Jones, P. F., Use of photoluminescence to investigate apparent suicides by firearms, *J. Forensic Sci.*, 26(2), 263–287, 1981.
- Louch, D., Motlagh, S., and Pawliszyn, J., Dynamics of organic compound extraction from water using liquid-coated fused silica fibers, *Anal. Chem.*, 64(10), 1187–1199, 1992.
- Lucas, A., *Forensic Chemistry and Scientific Criminal Investigation*, Edward Arnold & Co., London, U.K., 1948.
- Ludwig, N. and Wenz, W., Applications of focused ion beam systems in gunshot residue investigation, *J. Forensic Sci.*, 44(1), 105–109, 1999.
- MacCrehan, W. A., Smith, K. D., and Rowe, W. F., Sampling protocols for the detection of smokeless powder residues using capillary electrophoresis, *J. Forensic Sci.*, 43(1), 119–124, 1998.
- Mach, M. H., Pallos, A., and Jones, P. F., Feasibility of gunshot residue detection via its organic constituents, Part I: Analysis of smokeless powders by combined gas chromatography-chemical ionization mass spectrometry, *J. Forensic Sci.*, 23, 433–445, 1978.
- Mache, G. F., Thin-layer chromatographic/spectrophotometric analysis of certain components in aged double-base propellant, *J. Chromatogr.*, 38, 47–53, 1968.
- Maloney, R. S. and Thornton, J. I., Color tests for diphenylamine stabilizer and related compounds in smokeless powder, *J. Forensic Sci.*, 27, 318–329, 1982.
- Mann, M. J. and Espinoza E. O., The incidence of transient particulate gunshot primer residue in Oregon and Washington bow hunters, *J. Forensic Sci.*, 38(1), 23–27, 1993.

- Martens, C. S, Wesolowski, J. J., Kaifer, R., and John, W., Lead and bromine particle size distributions in the San Francisco Bay Area, *Atmos. Environ.*, 7, 905–914, 1973.
- Mathiesen, J. and Wood, W. G., Energy dispersive X-ray applications in forensic science, Rep. Q/M 29, Finnigan Corp., Sunnyvale, CA, 1973.
- Matricardi, V. R. and Kilty, J. W., The detection of primer residue particles using the scanning electron microscope with X-ray analysis, Pres. at the 28th Annu. Meet. of the Amer. Acad. of Forensic Sci., Washington, D.C., Feb. 1976.
- Matricardi, V. R. and Kilty, J. W., Detection of gunshot residue particles from the hands of a shooter, *J. Forensic Sci.*, 22(4), 725–738, 1977.
- Matricardi, V. R., Clark, M. S., and Defonja, F. S., The comparison of broken surfaces: a scanning electron microscope study, *J. Forensic Sci.*, 20, 507–523, 1975.
- Mauro, A. and Falso, G., Identification of ammunition used in a lethal robbery — comparison between scanning electron microscopy energy dispersive X-ray analysis and instrumental neutron activation analysis INAA measurements, *J. Forensic Sci.*, 38, 1237–1242, 1993.
- McCrone, W. C. and Delley, J. G., *The Particle Atlas*, Vol. 2, 2nd ed., Electron. Microsc. Atlas, Ann Arbor Science Publisher, Inc., Ann Arbor, MI, 1973.
- McFarland, R. C. and McLain, M. E., Rapid neutron activation analysis for gunshot residue determination, *J. Forensic Sci.*, 18(3), 226–231, 1973.
- McLain, J. H., *Pyrotechnics from the View Point of Solid State Chemistry*, Franklin Institute Press, 1980, 144–147, 192–193, 208–209.
- McQuillan, J., Detection of firearms discharge residues, *Anal. Proc.*, 20, 548–549, 1983.
- McQuillan, J., Firearms discharge residues sampling techniques, 10th Conf. of the Int. Assoc. of Forensic Sci., Oxford, U.K., 1984.
- Meier, J., Establishing when a shot was fired, *Int. Crim. Police Rev.*, 234, 22–26, 1970.
- Mendis, M. A. J., Sri Lankan shot pistols and ballistics, *Sci. Justice*, 37, 259–263, 1997.
- Meng, H. H. and Caddy, B., Fluorescence of ethyl centralite in gunshot residues, *J. Forensic Sci.*, 39(5), 1215–1226, 1994.
- Meng, H. H. and Caddy, B., Detection of n,n'-diphenyl-n'-diethylurea (ethyl centralite) in gunshot residues using high performance liquid chromatography with fluorescence detection, *Analyst*, 120, 1759–1762, 1995.
- Meng, H. H. and Caddy, B., High-performance liquid chromatographic analysis with fluorescence detection of ethyl centralite and 2,4-dinitrotoluene in gunshot residues after derivatization with 9-fluorenylmethylchloroformate, *J. Forensic Sci.*, 41, 213–220, 1996.
- Meng, H. H. and Caddy, B., Gunshot residue analysis — A review, *J. Forensic Sci.*, 42(4), 553–570, 1997.
- Mershon, W. J., Powers, Jr. W. H., Lentz, H. P., and Boughman, G. D., Application of microimaging to computer-controlled scanning electron microscopy, 1988 Joint Meet. of the Electron Microsc. Soc. of Amer., Microbeam Anal. Soc., and Microscopical Soc. of Can., Milwaukee, WI, August 7–12, 1988.

- Messler, H. R. and Armstrong, W. R., Bullet residue as distinguished from powder pattern, *J. Forensic Sci.*, 23, 687–692, 1978.
- Meyer, R., Explosives, *Verlag Chemie*, 4th rev. ed., Weinheim, 1993.
- Meyer, V. R., *Practical High Performance Liquid Chromatography*, 2nd ed., John Wiley & Sons, New York, 1994.
- Meyers, R. E. and Kopec, R. J., Identification of .22 calibre rimfire ammunition suitable for gunshot residue determination, *Identification News*, Sept. 10–13, 1976.
- Midkiff, Jr. C. R., *J. Police Sci. Admin.*, 3(1), 77–83, 1975.
- Miyauchi, H., Kumihashi, M., and Shibayama, T., The contribution of trace elements from smokeless powder to post firing residues, *J. Forensic Sci.*, 43(1), 90–96, 1998.
- Mogami, K. and Fukuda, K., Particle analysis for the detection of gunshot residue using a scanning electron microscope with EDX, Reports
- Mosher, P. V., McVicar, M. J., Randall, E. D., and Su, E. H. Gunshot residue — similar particles produce fireworks, *Can. Soc. Forensic Sci. J.*, 31(2), 157–168, 1998.
- Moulds, D. E., Antimony, *Miner. Facts Probl.*, 66–74.
- Munder, A., Chesler, S. N., and Wise, S. A., Capillary supercritical fluid chromatography of explosives: investigations on the interactions between the analytes, the mobile phase and the stationary phase, *J. Chromatogr.*, 521, 63–70, 1990.
- Munder, A., Christensen, R. G., and Wise, S. A., Microanalysis of explosives and propellants by on-line supercritical fluid extraction/chromatography with triple detection, *J. Microcolumn.*, 3, 127–140, Sept. 1991.
- Nag, N. K. and Sinha, P., A note on assessibility of firing distance from gunshot residues, *Forensic Sci. Int.*, 56, 1–17, 1992.
- Nesbitt, R. S., Wessel, J. E., and Jones, P. F., Conclusive detection of gunshot residue by the use of particle analysis, Rep. ATR-75 (7915)-2, The Aerospace Corporation, El Segundo, CA, 1974.
- Nesbitt, R. S., Wessel, J. E., and Jones, P. F., Detection of gunshot residue by use of the scanning electron microscope, *J. Forensic Sci.*, 21, 595–610, 1976 (originally published as Report ATR-75 (7915)-2, The Aerospace Corp. 1974).
- Nesbitt, R. S., Wessel, J. E., and Jones, P. F., Gunshot residue detection by scanning electron microscopy, Pres. at the Amer. Acad. of Forensic Sci. Meet., Washington, D.C., Feb. 1976.
- Newton, J., *Extractive Metallurgy*, John Wiley & Sons, New York, 1959, 282, 499.
- Newton, N. A. and Booker, J. L., The identification of smokeless powders and their residues by pyrolysis gas chromatography, *J. Forensic Sci.*, 24, 87–91, 1979.
- Niewohner, L. and Wenz, H. W., Applications of focused ion beam systems in gunshot residue investigation, *J. Forensic Sci.*, 44(1), 105–109, 1999.
- Northrop, D. M., Martire, D. E., and MacCrehan, W. A., Separation and identification of organic gunshot and explosive constituents by micellar electrokinetic capillary electrophoresis, *Anal. Chem.*, 63, 1038–1042, 1991.

- Northrop, D. M. and MacCrehan, W. A., Sample collection, preparation, and quantitation in the micellar electrokinetic capillary electrophoresis of gunshot residues, *J. Liq. Chromatogr.*, 15(6), 1041–1063, 1992.
- Northrop, D. M. and MacCrehan, W. A., Smokeless powder residue analysis by capillary electrophoresis, *NIJ Rep.* 600–691, Feb. 1997.
- Northrop, D. M., McCord, B. R., and Butler, J. M., Forensic applications of capillary electrophoresis, *J. Cap. Elec.*, 1(2), 158–168, 1994.
- Ong, C. P., Chin, K. P., Lee, H. K., and Li, S. F. Y., Optimization of supercritical fluid chromatographic separation of nitroaromatics by the overlapping resolution mapping scheme, *J. High Res. Chromatogr.*, 14, 249–253, 1991.
- Orloff, J., High-resolution focused ion beams, *Rev. Sci. Instrum.*, 64(5), 1105–1130, 1993.
- Owens, A. D., A re-evaluation of the aerospace corporation final report on particle analysis — when to stop searching for gunshot residue (GSR)? *J. Forensic Sci.*, 35, 698–705, May 1990.
- Parihar, D. B., Sharma, S. P., and Verma, K. K., Rapid estimation of explosive nitrates, *J. Chromatogr.*, 31, 551–556, 1967.
- Parker, C. E., Voyksner, R. D., Tondeur, Y., Henion, J. D., Harvan, D. J., Hass, J. R., et al., Analysis of explosives by liquid chromatography-negative ion chemical ionization mass spectrometry, *J. Forensic Sci.*, 27, 495–505, 1982.
- Particle Recognition and Characterization Program (PRC), *Operation Manual for Use with the TN-50 X-ray Analyzer*, Tracor Northern, WI, 19, 1984.
- Peak, S. A., A thin-layer chromatographic procedure for confirming the presence and identity of smokeless powder flakes, *J. Forensic Sci.*, 25, 679–681, 1980.
- Pillay, K. K. S., Jester, W. A., and Fox, H. A., New method for the collection and analysis of gunshot residue as forensic evidence, *J. Forensic Sci.*, 19(4), 768–783, 1974.
- Pimpton, G., *Fireworks: A History and Celebration*, Doubleday and Co., New York, 1984.
- Price, G., Firearms discharge residues on hands, *J. Forensic Sci. Soc.*, 5, 199–200, 1965.
- Price, G., Recent advances in ballistics laboratory methods, *J. Forensic Sci.*, 8(2,3), 83–90, 1968.
- Prime, R. J. and Kerbs, J., The analysis of ethylene glycol mononitrate and monomethylamine nitrate from commercial blasting agents in postblast samples, *Proc. of the Int. Symp. on Anal. and Detection of Explosives*, FBI Academy, Quantico, VA, 1983, 285.
- Quinn, C. C., Cartridge discharge residue contamination – the search for the source, *Sci. Justice*, 38, 81–84, 1998.
- Rabl, W. and Markwalder, C., Gunshot injury caused by a training bullet, *Arch Kriminol.*, 190(3–4), 88–96, 1992.
- Ravreby, M., Analysis of long-range bullet entrance holes by atomic absorption spectrophotometry and scanning electron microscopy, *J. Forensic Sci.*, 27, 92–112, 1982.

- Reed, G. E., McGuire, P. J., and Boehm A., Analysis of gunshot residue test results in 112 suicides, *J. Forensic Sci.*, 35, 62–68, 1990.
- Renfro, W. B. and Jester, W. A., Collection and activation analysis of airborne gunshot residues, *J. Radioanal. Chem.*, 15, 79–85, 1975.
- Renshaw, G. D., The estimation of lead, antimony and barium in gunshot residues by flameless atomic absorption spectrophotometry, CRE Rep. No. 103, Home Office Central Research Establishment, Aldermaston, U.K., 1973.
- Renshaw, G. D., Pounds, C. A., and Pearson, E. F., The quantitative estimation of lead, antimony and barium in gunshot residues by non-flame atomic absorption spectrophotometry, *Atomic Absorption Newslett.*, 12(2), 55–56, 1973.
- Ruch, R. R., Buchanan, J. D., Guinn, V. P., Bellenca, S. C., and Pinker, R. J., Neutron activation analysis on scientific crime detection — some recent developments, *J. Forensic Sci.*, 9, 119–133, 1964.
- Ruch, R. R., Guinn, V. P., Pinker, R. J., *Nucl. Sci. Eng.*, 20, 381, 1964.
- Rudzitis, E., Kopina, M., and Wahlgren, M., Optimization of firearm related residue detection by neutron activation analysis, *J. Forensic Sci.*, 18 (2), 93–100, 1973.
- Saferstein, R., *Criminalistics — An Introduction to Forensic Science*, Prentice-Hall, Englewood Cliffs, NJ, 1981, 347.
- Schlesinger, H. L., Lukens, H. R., Guinn, V. P., Hackleman, R. P., and Korts, R. F., Spec. Rep. on Gunshot Residues Measured by Neutron Activation Analysis, Gulf General Atomic, Inc., Rep. No. GA-9829, U.S. Atomic Energy Commission, San Diego, CA, Aug. 1990.
- Schmitz, J., Experience with the method of autoradiography for firing distance determination, *J. Radioanal. Chem.*, 15(1), 219–228, 1973.
- Schwartz, R. H. and Zona, C. A., A recovery method for airborne gunshot residue retained in human nasal mucus, *J. Forensic Sci.*, 40, 659–661, 1995.
- Schwoeble, A. J., Dalley, A. M., Henderson, B. C., and Casuccio, G. S., Computer-controlled SEM and microimaging of fine particles, *J. Metals*, August, 11–14, 1988.
- Scott, H. D., Coleman, R. F., and Cripps, F. H., Investigation of firearms discharge residues, Rep. 0-5/66, Atomic Weapons Research Establishment, Aldermaston, U.K., 1966.
- SEM/MPA Firearms Discharge Residues*, Vols. 1 and 2, Metropolitan Police Forensic Science Laboratory, London, U.K., 104–141, Nov. 1980.
- Sibert, R. W., Drugfire: Revolutionizing forensic firearms identification and providing the foundation for a national firearms identification network, <http://unojust.mitre.org/institutes/nij/documents/journals/drugfire/>, World Wide Web, 1996.
- Sild, E. H. and Pausak, S., Forensic applications of SEM/EDX, *Scanning Electron Microsc.*, 2, 185–192, 1979.
- Singer, R. L., David, D., and Houck, M. M., A survey of gunshot residue analysis methods, *J. Forensic Sci.*, 41(2), 195–198, 1996.
- Sinha, J. K., Time of firing of shot shells, *J. Forensic Sci.*, 21(1), 171–175, 1976.

- Sinha, J. K. and Misra, G. J., Detection of powder particles at the crime scene, *J. Forensic Sci.*, 16, 109–111, 1971.
- Slavin, W. and Carnrick, G. R., A survey of applications of the stabilized platform furnace and Zeeman correction, *Atmos. Spectrosc.*, 6(6), 157–160, 1985.
- Smith, C., Spherical coordinates for plotting the positions of powder residues, *J. Forensic Sci.*, 18(2), 101–109, 1973.
- Steinberg, M., Leist, Y., Goldschmidt, P., and Tassa, M., Spectrophotometric determination of nitrites in gunpowder residue on shooters' hands, *J. Forensic Sci.*, 29, 464–470, 1984.
- Stone, I. C., Observations and statistics relating to suicide weapons, *J. Forensic Sci.*, 32(3), 711–716, 1987.
- Stone, I. C., Jr. and Petty, C. S., Examination of gunshot residue, presented at the AAFS 26th Annu. Meet., Dallas, TX, Feb. 1974.
- Sugarman, L. A., The concentration and isolation of gunshot residues for particle analysis, *J. Forensic Sci. Soc.*, 27, 135, 1987.
- Tassa, M. and Zeldes, N., Characterization of gunshot residue particles by localizations of their chemical constituents, *Scanning Electron Microsc.*, 2, 175–178, 1979.
- Tassa, M., Adan, N., Zeldes, N., and Leist, Y., A field kit for sampling gunshot residue particles, *J. Forensic Sci.*, 27(3), 671–676, 1982.
- Taylor, R. L., Taylor, M. S., and Noguchi, T. T., *Scanning Electron Microscopy*, Ill. Inst. of Tech. Res. Inst., Chicago, IL, 1975, 494–500.
- Tebbett, I., Ed., *Gas Chromatography in Forensic Science*, Ellis Horwood, New York, 1992.
- Terabe, S., Otsuka, K., Ichikawa, K., Tsuchiya, A., and Ando, T., Electrokinetic separations with micellar solutions and open-tubular capillaries, *Anal. Chem.*, 56, 111–113, 1984.
- Tewari, S. N., A new micro-chemical reagent for detection of gunpowder residues in shooting cases, *Int. Crim. Pol. Rev.*, 281, 220, 1974.
- Thornton, J. I., Close proximity gunshot residues, *J. Forensic Sci.*, 31, 756–757, 1986.
- Tillman, W. L., Automated gunshot residue particle search and characterization, *J. Forensic Sci.*, 32(1), 62–71, 1987.
- Torre, C. and Gino, S., Epidermal cells on stubs used for detection of GSR with SEM-EDX: analysis of DNA polymorphisms, *J. Forensic Sci.*, 41(4), 658–659, 1996.
- Touchstone, J. C., Ed., *Techniques and Applications of Thin Layer Chromatography*, John Wiley & Sons, New York, 1985.
- Trowell, J. M., Gas chromatographic determination of nitrated derivative of glycerin in aged double based propellants, *Anal. Chem.*, 42, 1440–1442, 1970.
- Trowell, J. M. and Philpot, M. C., Gas chromatographic determination of plasticizers and stabilizers in composite modified double-base propellants, *Anal. Chem.*, 41, 166–168, 1969.
- Tschirhart, D. L., Noguchi, T. T., and Klatt, E. C., A simple histochemical technique for the identification of gunshot residue, *J. Forensic Sci.*, 36(2), 543–547, 1991.

- Twibell, J. D., Home, J. M., Smalldon, K. W., and Higgs, D. G., Transfer nitroglycerine to hands during contact with commercial explosives, *J. Forensic Sci.*, 27, 783–791, 1982.
- Twibell, J. D., Home, J. M., Smalldon, K. W., Higgs, D. G., and Hayes, T. S., Assessment of solvents for the recovery of nitroglycerine from hands using cotton swabs, *J. Forensic Sci.*, 27, 792–800, 1982.
- Twibell, J. D., Wright, T., Sanger, D. G., Bramley, R. K., Lloyd, J. B. F., and Downs, N. S., The efficient extraction of some organic explosives from hand swabs for analysis by gas liquid and thin-layer chromatography, *J. Forensic Sci.*, 29, 277–283, 1984.
- Urbanski, T., *Chemistry and Technology of Explosives*, Vol. 3, Pergamon Press, Elmsford, New York, 1967.
- Via, J. C. and Taylor, L. T., Chromatographic analysis of nonpolymeric single base propellant components, *J. Chromatogr. Sci.*, 30, 106–110, 1992.
- Voskertchian, G. P. and Pavilova, G. V., The possibility of determining the time of shooting by ESR spectroscopy, presented at the 41st Annu. Meet. of the Amer. Acad. of Forensic Sci., Las Vegas, NV, 1989.
- Voskertchian, G. P. and Pavilova, G. V., Spectrophotometric determination of time since discharge of firearms, *Crime Lab. Dig.*, 22(1), 5–10, 1995 and *AFTE J.* 26(3), 216–222, 1994.
- Vouros, P., Petersen, B. A., Colwell, L., and Karger, B. L., Analysis of explosives by high performance liquid chromatography and chemical ionization mass spectrometry. *Anal. Chem.*, 49, 1039–1044, 1977.
- Voyksner, R. D. and Yinon, J., Trace analysis of explosives by thermospray high-performance liquid chromatography-mass spectrometry, *J. Chromatogr.*, 354, 393–405, 1986.
- Walker, J. T., Bullet holes and chemical residues in shooting cases, *J. Crim. Law Criminol.*, 31, 497–519, May-June 1940, Mar-Apr 1941.
- Wallace, J. S., Discharge residue particles from blank cartridges, *AFTE J.*, 18(4), 33–38, 1986.
- Wallace, J. S., Discharge residue from mercury fulminate-primed ammunition, *Sci. Justice*, 38, 7–14, 1998.
- Wallace, J. S. and Keeley, R. H., A method for preparing firearms residue samples for scanning electron microscopy, *Scanning Electron Microsc.*, 2, 179–184, 1979.
- Wallace, J. S. and McKeown, W. J., Sampling procedures for firearms and/or explosives residues, *J. Forensic Sci. Soc.*, 33, 107–116, 1993.
- Wallace, J. S. and McQuillan, J., Discharge residue from cartridge-operated industrial tools, *J. Forensic Sci. Soc.*, 24(5), 495–508, 1984.
- Wang, C. Y., *Antimony*, 3rd ed., Charles Griffin & Company, London, 1952, 170.
- Ward, D. C., Gunshot residue collection for scanning electron microscopy, *Scanning Electron Microsc.*, 3, 1031–1036, 1982.
- Wessel, J. E. and Wolten, G. M., Symposium synopsis: detection of gunshot residue, Rep. ATR-76-(7915)-2, The Aerospace Corp., El Segundo, CA, 1975.

- Wessel, J. E., Jones, P. F., Kwan, Q. Y., Nesbitt, R. S., and Rattin, E. J., Gunshot residue detection, ATR-75 (7915)-1, The Aerospace Corp., El Segundo, CA, Sept. 1974.
- Wessel, J. E., Jones, P. F., Kwan, Q. Y., Nesbitt, R. S., and Rattin, E. J., Equipment systems improvement program gunshot residue detection, The Aerospace Corp., El Segundo, CA, 1974.
- Wessel, J. E., Nesbitt, R. S., and Jones, P. F., New techniques for the detection of gunshot residue, presented at the 7th Int. Meet. of Forensic Sci., Zurich, Switzerland, Sept. 1975.
- White, D. E., Antimony in the United States, *Mineral Investigations Res. Map MR-20*, U.S. Geol. Survey, 1962.
- White, R. S. and Gross, M. L., Deposition of gunshot residue at various distances from discharging firearms, presented at the SAFS Meet., Raleigh, NC, 1989.
- White, R. S. and Owens, A. D., Automation of gunshot residue detection and analysis by scanning electron microscopy/energy dispersive X-ray analysis (SEM/EDX), *J. Forensic Sci.*, 32, 321–330, 1987.
- Wilber, C. G., *Ballistic Science for the Law Enforcement Office*, Charles C. Thomas, Springfield, IL, 1977.
- Wolten, G. M. and Nesbitt, R. S., On the mechanism of gunshot residue particle formation, *J. Forensic Sci.*, 25(3), 533–545, 1980.
- Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., Equipment systems improvement program — final report on particle analysis for gunshot residue detection, Rep. ATR-77 (7915)-3, The Aerospace Corporation., Washington, D.C., Sept. 1977.
- Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., Particle analysis for the detection of gunshot residue. I: Scanning electron microscopy/energy dispersive X-ray characterization of hand deposits, *J. Forensic Sci.*, 24(2), 409–422, 1979.
- Wolten, G. M., Nesbitt, R. S., Calloway, A. R., Loper, G. L., and Jones, P. F., Particle analysis for the detection of gunshot residue. II: Occupational and environmental particles, *J. Forensic Sci.*, 24(2), 423–430, 1979.
- Wolten, G. M., Nesbitt, R. S., and Calloway, A. R., Particle analysis for the detection of gunshot residue. III: The case record, *J. Forensic Sci.*, 24(4), 864–869, 1979.
- Wood, W. G. and Mathiesen, J. M., *Trace Element Analysis by Energy Dispersive X-Ray Spectroscopy*, Finnigan Corp., Sunnyvale, CA, 1974.
- Woolever, C. A., Starkey, D. E., and Dewald, H. D., Differential pulse anodic stripping voltammetry of lead and antimony in gunshot residues, *Forensic Sci. Int.*, 102(1), 45–50, 1999.
- Wrobel, H. A. and Millar, J. J., Characterization of .22 calibre ammunition, *Scanning*, 15(3), 118–119, 1993.
- Wrobel, H. A., Millar, J. J., and Kijek M., Comparison of properties of adhesive tapes, tabs and liquids used for the collection of gunshot residue and other trace materials for SEM analysis, *J. Forensic Sci.*, 43(1), 178–181, 1998.

- Wrobel, H. A., Millar, J. J., and Kijek, M., Identification of ammunition from gunshot residue and other cartridge related materials — a preliminary model using .22 caliber rimfire ammunition, *J. Forensic Sci.*, 43(2), 324–328, 1998.
- Yasuda, S. K., Identification of impurities in α -trinitrotoulene by thin-layer chromatography, *J. Chromatogr.*, 13, 78–82, 1964.
- Yasuda, S. K., Identification of N-nitroso- and nitrodiphenylamine by two-dimensional thin-layer chromatography, *J. Chromatogr.*, 14, 65–70, 1964.
- Yellin, E., Padova, A., Even, O., Kaplan, M., and Almog, J., A new method for the determination of gunshot residue on hands, presented at the 7th Int. Meet. of Forensic Sci., Zurich, Switzerland, Sept. 1975.
- Yinon, J. and Hwang, D. G., High-performance liquid chromatography-mass spectrometry of explosives, *J. Chromatogr.*, 268, 45–53, 1983.
- Yinon, J. and Zitrin, S., *The Analysis of Explosives*, Pergamon Press, New York, 1981, 27.
- Zeichner, A. and Levin, N., Forensic applications of a combined SEM/EDX automated system for detection and identification of microscopic particles with special emphasis on gunshot residues, *Ultramicroscopy*, 32, 194, 1990.
- Zeichner, A., Levin, N., and Dvorachek, M., Gunshot residues formed by using ammunitions that have mercury fulminate based primers, *J. Forensic Sci.*, 37, 1567–1573, 1992.
- Zeichner, A. and Levin, N., Collection and efficiency of gunshot residue (GSR) particles from hair and hands using double sided adhesive tape, *J. Forensic Sci.*, 38, 571–84, 1993.
- Zeichner, A. and Levin, N., Casework experience of GSR detection in Israel, on samples from hands, hair and clothing using an autosearch SEM/EDX system, *J. Forensic Sci.*, 40(6), 1082–1085, 1995.
- Zeichner, A. and Levin, N., More on the uniqueness of gunshot residue (GSR) particles, *J. Forensic Sci.*, 41, 1027–1028, 1997.
- Zeichner, A., Foner, H. A., Dvorachek, M., Bergman, P., and Levin, N., Concentration techniques for the detection of gunshot residues by scanning electron microscopy/energy-dispersive X-ray analysis (SEM/EDX), *J. Forensic Sci.*, 34(2), 312–320, 1989.
- Zeldes, N. and Tassa, M., Conversion of existing SEM components to form an efficient backscattered electron detector, and its forensic applications, *Scanning Electron Microsc.*, 124, 155–158, 1979.
- Zhang, Z. and Pawliszyn, J., Headspace solid-phase microextraction, *Anal. Chem.*, 65(14), 1843–1852, 1993.