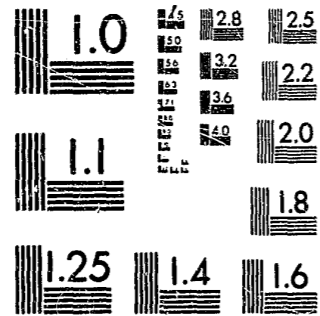


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First Year

Final Report

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010- Development of Techniques for the Detection of Airborne Gunshot Residues, final report

To

U.S. Department of Justice  
Law Enforcement Assistance Administration  
National Institute of Law Enforcement and Criminal Justice  
Washington, D.C. 20530

041- NILECS

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Foreword

W. F. Witzig, Professor and Head of  
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The goal of this work is to bring to bear the more sophisticated technologies such as nuclear activation analysis in the field of law enforcement. Success in this work will make available a field technique for trained enforcement personnel to rapidly detect the location and possibly the time that a firearm was discharged should airborne residue remain. While substantial progress has been made since the first crude tests of the authors, several important problems remain to be solved and ideas on how to solve them are being explored.

## Preface

This report marks the conclusion of the first year of a proposed two year program to develop a technique for the detection of airborne gunshot residue. The question to be answered is "Where and possibly when was the shot fired?" Over most of the first year a vacuum filter system was employed for residue collection. Recently an electrostatic precipitator was successfully employed for this collection. The analytical technique employed was the highly sensitive neutron activation analysis. The elements whereby the gunshot residue was quantitatively detected were antimony and barium. To date the technique has successfully detected gunshot residue from a single 22 caliber bullet up to 36 hours after firing.

The authors would like to thank the Director and Staff of the Penn State Breazeale Nuclear Reactor for their excellent support of this project. They also extend thanks to Captain L. D. Jenkins, Director of the Pennsylvania State Police Crime Laboratory, Sergeant James Deffley, Head of the Crime Laboratory's Ballistic Section, Dr. H. L. Guttentplan, Professor of Law Enforcement and Correction, Pennsylvania State University, and Dr. W. J. Moroz, Director of the University's Center for Air Environment Studies, for providing valuable technical guidance and criticisms during the course of this first year's program. The authors wish to thank the National Institute of Law Enforcement and Criminal Justice, U. S. Department of Justice, for granting the funds for the first half of this project.

## Summary

### Purpose

The major objective of this project is to develop a technique which can tell an investigative officer whether or not a firearm has recently been fired in a room, and if so, when it was fired. The technique to be developed is envisioned as operating in the following way: Upon arriving at the scene of a crime which involved the discharging of a firearm, the investigative officer would use a small portable air sampler to collect samples of the debris suspended in the air of the rooms potentially involved. The samples would then be promptly taken to a laboratory having a neutron activation analysis capability, where they would be rapidly analyzed for traces of gunshot residues. The presence of such residues would establish where the firearm was fired.

In order to establish when the weapon was fired, possibly a carefully staged reenactment of the firing would be conducted at a later date. This would be followed by periodic collections of the resulting airborne material to measure the rate at which the residue is removed from the air in the room, and thus establish the approximate time of the original firing.

The objectives of the first year's program were to demonstrate the feasibility of detecting airborne gunshot residues using neutron activation analysis and to evaluate the potential of obtaining time of firing information.

## Scope

It was anticipated that two years would be required to develop the techniques to the point where they could be tested on actual cases. The scope of the first year's effort was given in the original proposal as follows:

1. Testing of various collecting systems and filter media for the recovery of gunshot residues suspended in air.
2. Establishment of optimum activation analysis methods for the detection of gunshot residues.
3. Development of correct methods of handling and transporting the collected residues to avoid sample contamination.
4. Exploration of the potential of obtaining time of firing information by conducting tests in a closed room and then employing the techniques in 1, 2, and 3 above.

In the preliminary stages of this program, a test room was constructed for gunshot firing and subsequent sample collection.

The basis for most tests run during the first year's program was the collection of airborne residue from the discharge of a single 22 caliber bullet in the test room.

## Methods

The weapon involved in the initial tests during the first year was an E & R nine shot revolver. In an effort to obtain reproducibility, the same revolver chamber was employed for most of the tests. The ammunition was all taken from the same batch of western 22 caliber bullets.

A standard air filter collection system was employed for most of the tests, consisting of a vacuum pump which drew the air of the test room through a 2 inch, 0.45 micron membrane air filter disk.

An electrostatic precipitator was also successfully employed in some of the later tests.

Air samples were collected as a function of time up to 36 hours after the time of firing. The collected samples were prepared for neutron activation in the Penn State Breazeale Nuclear Reactor. The irradiation conditions varied with the amounts of antimony and barium present in the samples.

After post-irradiation handling, the gamma spectra of the resulting radioisotopes were measured on a high resolution Ge(Li) detector with the resulting data being stored in a 1024 pulse height analyzer. The resulting accumulated spectral data was then transferred to magnetic tape for subsequent computer processing by the Penn State IBM 360 computer.

The intensity of the measured radioisotopes of antimony and barium as compared to standards gave information as to the amount of antimony and barium in the air filter sample.

## Findings and Conclusions

It has been demonstrated in this first year's project that the method being investigated can successfully detect gunshot residues up to at least 36 hours after the discharge of a single 22 caliber bullet into a still room.

The technique as it has been developed to date is not yet able to provide time of firing information.

The electrostatic precipitator collection system shows greater promise than air filter systems for collecting gunshot residues because of higher collection efficiency for a wider range of particle sizes and because of the ability to provide some particle discrimination according to particle size.

The second year's program will explore the merits of this type of collecting system.

Grantee's  
Report of Expenditures

The regulations of The Pennsylvania State University require that this report be submitted from the Office of the University's Vice President for Finance. It will therefore be submitted under separate cover shortly.

Text

Introduction

The concept for this project resulted from conversations between personnel of the Pennsylvania State Police Crime Laboratory and personnel from The Pennsylvania State University's Nuclear Engineering Department during the year 1969. The question being discussed was "Can a method be developed which could tell whether or not a firearm had been discharged in a room, and if so, when it was fired?" It was known from previous work by such investigators as Dr. V. P. Guinn of Gulf General Atomic, M. J. Pro of the Internal Revenue Service, and others that neutron activation analysis can be employed to determine whether or not an individual had fired a weapon. This can be accomplished by collecting and analyzing the debris on the individual's skin and clothing for the barium and antimony found in ammunition. However, this type of evidence can readily be destroyed by washing, and does not answer the question of where and when the weapon was discharged.

A second method might be the analysis of the debris deposited on the walls and floor immediately after firing or after subsequent settling. This method offers the potential of identifying not only in which room the weapon was fired, but where in the room. But it would not answer the question of when the weapon was fired. Also, it would require a complicated and systematic sampling of all suspected areas, and the sensitivity of the technique would be very much a function of the amounts of other material collected which would be unrelated to the firing event and which would interfere with the analysis.

The third approach, and the one which developed out of the above-mentioned conversations, was to take advantage of the gunshot residues remaining in the air to answer these questions. This concept became the basis for the project herein described.

#### Methods

##### A. Nature of Airborne Gunshot Residues

Airborne gunshot residues result primarily from two sources:

1. The combustion of gun powder and primer,
2. The melting and vaporization of the lead bullet slug.

The size of particles resulting from combustion range from 0.03 to 0.3 microns; the size of particles from the vaporization of lead are probably in the same range. Most other types of commonly occurring airborne residues are larger than this, including lint (10 microns and larger), dust (0.1 microns and larger), and pollen (10 microns and larger). Particles as small as those formed in the discharge of a firearm should remain suspended in air for many days with their removal resulting from the agglomeration of the smaller particles to form larger particles which will settle out under the influence of gravity or from their being exhausted from the area by convective air currents.

Previous studies by Guinn, et al.\* at Gulf General Atomic indicated that of the elemental constituents of the residues produced from discharging the major brands of ammunition, the elements with the greatest neutron activation sensitivity were antimony and barium. They attributed the source of the barium to the primer of the bullet, while the antimony came from the primer and from the bullet lead, where it is used as a hardening agent.

##### B. Equipment and Facilities

###### 1. Test Room

A test room was designed and constructed for discharging test weapons with subsequent controlled sampling of the suspended gunshot residues. It was designed with six basic features:

- a. Isolation from the external environment
- b. Low cost
- c. Ease of dismantling and rebuilding in the event the room became contaminated with the suspended residues
- d. Controlled ventilation of the room during tests
- e. Complete exhaustion of the room at the end of a test
- f. Access to the firearms and air sampling equipment without disturbing the room environment.

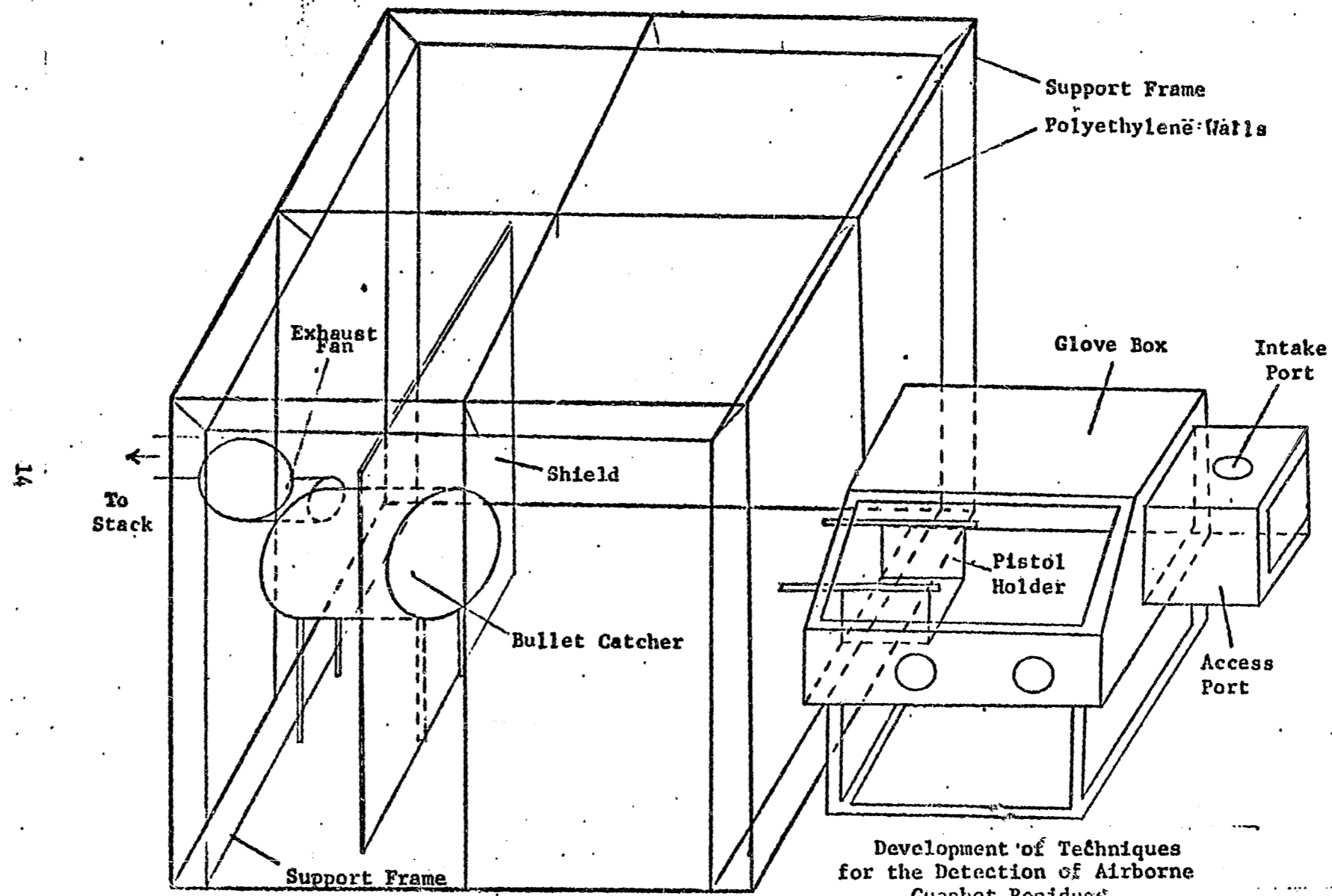
\* Applications of Neutron Activation Analysis in Scientific Crime Investigation, AEC Report GA-9822, January 21, 1970.

The testing facility design arrived at is shown in Figure 1. It consists of a large room constructed from polyethylene sheeting and sealed at the edges so that there is no venting at the corner seams. This polyethylene room is suspended from an aluminum frame which is adjustable to accommodate a room as large as a 12 foot cube. A polyethylene-walled room was chosen so that whenever sufficient removal of all suspended and settled gunshot residues cannot be accomplished by ventilation, the room can be removed from its supporting frame and collapsed. The polyethylene can be sent to solid waste handling and a new room constructed from clean polyethylene sheeting.

Access to the polyethylene room is obtained through a K.S.E. controlled atmosphere glove box from which the left side has been removed to provide a suitable access for the firearm and airborne residue collection samplers. The access port of the glove box allows the insertion and removal of firearms, equipment, and samples during testing without affecting the controlled conditions in the testing room. An aluminum frame was built and bolted to the glove box where it opens into the polyethylene room. A carriage to which the pistol is bolted rides on this frame. This carriage allows the pistol to be slid forward into the test room for firing and slid back into the glove box for loading and unloading. The U-bolts which clamp the pistol to the carriage are adjustable so that the alignment of the pistol barrel in relationship to the bullet catcher can be properly set.

A satisfactory bullet catcher was constructed from a steel 30 gallon drum, 14 inches in diameter by 22 inches long, packed with cotton padding. This catcher is mounted on a steel frame to obtain a proper height in relation to the pistol mount in the glove box. The bullet catcher was tested on an outdoor firing range using a 22 caliber revolver, a 22 caliber rifle, and a 45 caliber automatic revolver. Nine shots from a 22 caliber revolver were fired at a point-blank range into the same point of the center of the bullet catcher. The deepest penetration of the cotton padding was about 16 inches. There was some evidence of a "tunneling" effect due to the repeated firings in the same area. The bullets stopped within two inches of each other, and in this area the cotton padding became significantly more compressed, increasing the penetration resistance to subsequent bullets. It therefore appears that repeated firings can be employed without any danger of a bullet escaping the bullet catcher. The bullets from the 22 caliber rifle penetrated about 19 inches of padding while the bullets from the 45 caliber revolver penetrated between 17 and 18 inches of padding. Thus the bullet catcher was demonstrated to be suitable not only for stopping bullets from the 22 caliber pistol but also for these more powerful weapons.

To eliminate the remote possibility of a ricochet back toward the glove box where personnel would be located during test firing, a 1-1/2 inch thick plywood screen was constructed from two sections of plywood, dimensions 3/4 inch by 6 feet by 4 feet.



Development of Techniques  
for the Detection of Airborne  
Gunshot Residues

Figure 1



This screen is positioned at the entrance to the bullet catcher with its plane perpendicular to the bullet projection. A 14 inch diameter hole is cut into the screen such that the screen surrounds but does not cover the opening to the bullet catcher. Thus, if a bullet is somehow off target and should glance off the side of the bullet catcher, or should penetrate the bullet catcher and ricochet off the concrete wall in back of the bullet catcher, it would be stopped by the plywood screen before reaching the area where testing personnel would be located.

Ventilation of the room during testing, when required, and for purging the room of suspended gunshot residues at the end of testing, is accomplished by a floor-mounted Marshall Ventilating Exhauster fitted with two dampers to control the ventilation rate. During ventilation, fresh air flows through the glove box into the polyethylene room, and then exhausts into a ducting system. The exhaust system is composed of 110 feet of six inch ducting. This transports the exhaust air from the polyethylene room to a location outside the Reactor Facility where it empties at ground level at a point approximately 50 feet from the building. Smoke tests indicate that at this location the prevailing winds will blow any remaining suspended residues away from the building. Such ventilation procedures minimize the contamination of the glove box with gunshot residues and keep the air entering the glove box free of previously exhausted residues.

The revolver used in the testing was a 22 caliber H & R revolver, nine shot model. Western, T-22, standard velocity ammunition was used. An H & R 32 caliber revolver was used for one test. For this test, the revolver was hand-held. Arrangements have been made with the Pennsylvania State Police for the use of a 38 caliber service revolver with a six inch barrel for the testing of larger caliber firearms.

## 2. Air Sampling Equipment

Four devices for particulate air sampling were tested during the year. They are:

- a. Gelman Hurricane Air Sampler
- b. Fisher Vacuum Pump with a General Electric 1/6 horsepower motor and a 2 inch filter holder
- c. Gelman Vacuum Pump (1/3 horsepower) Model 1034-H10 and a 2 inch filter holder
- d. Bendix electrostatic precipitator

The Hurricane air sampler is a high volume device used to sample large areas/for gross (greater than 5 micron particle size) particles. It is light and simple in operation. However, mechanical breakdown resulted from the extended high pressure operation required with the smaller pore size filter media.

The vacuum pump systems with filter holders were found to be suitable for collecting the finer sized particles (.1 to 5 microns). They are designed to maintain the high pressure differential needed to give an adequate flow rate when using the finer filters.

The electrostatic precipitator combines efficient collection of small particles with a large sampling rate. It can be battery operated, which extends its flexibility. A cyclone-pre-separator can be employed to screen out particles larger than about 10 microns before they reach the precipitator.

### 3. Filter Media Employed

About 15 types of air filter media were tested to find the best combination of collection efficiency, handling properties, freedom from interference activities, and ease of radiochemical processing. The Gelman GA-6 triacetate metricel membrane filter having a 0.45 micron pore size was chosen as having the best combination of the above-mentioned properties. This filter paper, used with the Fisher vacuum pump and filter holder, was chosen as the basis for repetitive testing and the norm to which other sampling equipment and filter media were compared.

Whatman #50 filter paper was employed for use in the electrostatic precipitator because it had been used in previous neutron activation analysis tests, and was readily available. This collection media is placed as a coiled cylinder in the unit's sampling tube which can be capped at both ends before and after sample collection to avoid sample contamination.

### 4. Breazeale Nuclear Reactor

The Pennsylvania State University's Breazeale Nuclear Reactor was employed for all neutron irradiations conducted in this project. The TRIGA type reactor can operate in the steady-state mode at a power level of 1 megawatt with a maximum neutron flux of  $3 \times 10^{13}$  neutrons/cm<sup>2</sup>-sec, or it can be pulsed to a peak power of 2000 megawatts with a maximum neutron flux of  $6 \times 10^{16}$  neutrons/cm<sup>2</sup>-sec. This last feature is used for pulse neutron activation analysis involving radioisotopes with half-lives as short as a few seconds. The reactor core, suspended from a moveable bridge, can be positioned in the "swimming pool" to provide the most effective experimental setup. Special equipment directly associated with the reactor includes two thermal columns, pneumatic "rabbit" tubes and several beam ports.

Both in-core and pneumatic "rabbit" irradiations were utilized in this project.

### 5. Gamma Ray Spectroscopy Facilities

The Radionuclear Applications Laboratory is in the research wing of the Nuclear Reactor Facility. It consists of a radiochemistry laboratory and a large counting laboratory. Besides the usual chemical facilities, the Laboratory is equipped with a 1024 Channel Nuclear Data Model 2200 Pulse Height Analyzer with a 4096 ADC. It also has a 100 channel TMC Gamma Scope which is primarily employed as part of a flux monitoring system, along with a 3 inch diameter by 3 inch thick NaI(Tl) detector. A thin NaI(Tl) detector is employed for x-ray spectroscopy. A large low background shield houses a 4 inch diameter by 3 inch thick NaI(Tl) detector for high efficiency radiation detection. A second crystal of the same size is employed with the first crystal for coincidence counting measurements.

Two high resolution Ge(Li) detectors are available for use in this project. One is in the form of a circular cylinder having a volume of approximately 30 cubic centimeters. The other is in the form of a circular disk 0.5 centimeters thick with a volume of 2 cubic centimeters. For x-rays and low energy gammas of energy lower than 100 kev, the thin detector has somewhat better detection efficiency and a considerable reduction in interference from high energy activation products of the dust and filters. For the higher energy gammas, the larger detector has a considerably higher detection efficiency.

## 6. Data Processing Facilities

The Pennsylvania State University's IBM System/360 Model 67 is employed for data processing. A recently acquired direct access terminal, Model #2741, located at the reactor facility, is being employed to speed up the turn-around time in computer processing of a data run.

Two computer codes entitled "Energy" and "PTT" are part of the data processing programs of the Radionuclear Laboratory. These programs have been modified and adapted for use in analyzing the gamma-ray spectral data obtained from the high resolution radiation detection instrumentation for antimony and barium.

### C. Evaluations of Air Filters and Filter Media

#### 1. Air Sampler Evaluations

From the tests made at the start of this program, it is possible to evaluate the three sampling systems used as to their value as gunshot residue collection systems. The criteria used in their evaluation considered:

- a. Gunshot residue collection efficiency
- b. Portability and flexibility
- c. Filter handling and neutron activation characteristics

The Fisher Vacuum Pump system was found to be the best air filter system tested for preliminary testing, and became the standard system with which the other systems were compared. It was designed for high pressure differentials, and could be used in fine particle sampling using 0.45 micron filters. However, the flow rate was very low, necessitating long sampling times. It required a 110 volt power supply, which severely limits its portability for subsequent field testing.

The Gelman Hurricane Air Sampler is designed for high flow rates and low pressure differentials. The use of a fine filter for gunshot particle collection caused a high pressure differential to arise, and since the equipment was not designed for this high pressure differential, rapid mechanical wear resulted. Although this unit is light, it also requires a 110 volt power supply. The exhaust of the sampler also introduces collection errors in that it tends to stir up settled dust particles. Thus it was found to be unsuitable for preliminary testing.

The 0.45 micron filter was allowing most of the gunshot residue to pass through the system without being collected. When an effort was made to use the vacuum pump collection system to collect the smaller particles by using 0.1 micron pore size filters, the amount of air throughout was greatly reduced, producing samples containing very little residue. To solve the problem of obtaining high collection rates of very fine particles, the authors have turned to the consideration of electrostatic precipitators.

The electrostatic precipitator system tested recently appears to be clearly superior to the vacuum pump systems tested. As it collects particles by electrostatic precipitation, no pressure differential is required across a filter, and a high flow rate can be used. It is extremely effective for collecting small particles (below 0.01 microns) and the particles are generally deposited according to particle size along the length of the collecting cylinder. This allows for the possible collection of selective portions of the airborne gunshot residues with less interference from the larger dust particles. The unit is very light, and can be battery operated without loss of efficiency.

It was discovered that cigarette smoke would deposit itself on the same region of the collection media as the airborne gunshot residues, indicating that both of these products of combustion produce particles of about the same range of sizes. This finding has led to a rapid method of calibrating an electrostatic precipitator for gunshot residue collection. The smoke rapidly leaves a brown ring on the paper at the location where the investigator should look for the maximum concentration of airborne gunshot residue.

## 2. Filter Media Evaluations

The four parameters used in evaluating the filter media were:

### a. Low neutron activation interference by the filter media

Since it would be extremely difficult to separate the collected airborne gunshot residue from the filter material for neutron activation analysis, it must not contain any significant amounts of elements which, upon irradiation, will produce radioisotopes whose gamma spectra will interfere with those of the antimony and barium radioisotopes activated in the gunshot residue.

### b. High efficiency for the collecting of airborne gunshot residue

The gunshot residues are suspected to lie in a size range of 0.03 to 0.3 microns. A filter must then be used which is fine enough to collect these small particles. However, the flow rate through the filters should not be so low that it necessitates long sampling times.

### c. Physical strength

Since the ultimate objective of this project is to have a method which can be used routinely in field applications, the ability of the filters to be easily handled without tearing, cracking, or curling is an important characteristic.

### d. Sample preparation

The filter must be able to be transformed into a reproducible geometry. This may be done either by chemical reduction, dissolution, or simple folding.

Table 1 summarizes the results of several of the filter paper types studied most extensively. A total of 12 other types of filter papers were also studied. The standard filter paper type was the GA (triacetate metricel). The Nucleopore filter has somewhat better properties, but these were not available until the end of this first year's project.

Since the collection paper on the electrostatic precipitator has particles deposited on it rather than filtering through it, the collection efficiencies and flow rate parameters are not important. It still must have good neutron activation, strength, and sample preparation characteristics. An additional requirement is introduced in that the filter media must be rigid enough to stay rolled in the sample collection tube without falling out. Work has been done with Whatman #50 filters in other experiments, and they have excellent handling and neutron activation characteristics. They are readily available, and so were used in the tests. The sample preparation area has not yet been developed, but modifications from other applications should be possible.

Table 1

Filter Paper Characteristics

| Filter                      | Neutron Activation Characteristics | Collection Efficiency | Flow Rate | Strength  | Sample Preparation |
|-----------------------------|------------------------------------|-----------------------|-----------|-----------|--------------------|
| GA(triacetate metricel)     | Good                               | Good                  | Fair      | Fair      | Good               |
| VM(vinyl metricel)          | Poor                               | Good                  | Fair      | Excellent | Good               |
| AN(acrylonitrile copolymer) | Poor                               | Good                  | Good      | Excellent | Good               |
| Nucleopore (polycarbonate)  | Excellent                          | Good                  | Excellent | Excellent | Good               |
| Whatman 50                  | Excellent                          | NI                    | NI        | Excellent | NS                 |

NI Not important  
 NS Not studied to date

23

#### D. Techniques for Sample Collection, Handling, and Analysis

##### 1. Pistol Firing and Sample Collection Procedures

A detailed firing procedure was developed to prevent accidents during a firing test. This procedure, given in Appendix A, requires notification of reactor personnel that a test is to be made, signs to be posted at all entrances, and a visual check to be made prior to loading the revolver. All personnel observing the test stand behind the glove box and are protected by the same bullet shield that protects the experimenter. Ear protectors are used to prevent injury due to an experimenter's presence at repeated firings.

To limit the number of variables involved in this initial phase of the project, the following conditions were employed in most test firings:

- a. A still room environment was employed, i.e., no ventilation of the room during the test.
- b. Only one 22 caliber bullet was fired per test. This was considered to be the condition depositing the minimum amount of gunshot residue into the air. The bullets were all taken from the same batch of western-22 ammunition.
- c. The same H & R nine shot revolver was employed for all 22 caliber bullet firings and in most tests the same revolver chamber was employed.
- d. Although a variety of sampling times and intervals was employed covering a period up to 36 hours after firing, a standard set of sampling procedures was employed for establishing the degree of reproducibility in the amount of residue being deposited per shot. These sampling conditions are as follows:

Start of Sample  
Collection after Firing

Sampling  
Time

Hours

|    |        |
|----|--------|
| 0  | 15 min |
| 4  | 15     |
| 8  | 30     |
| 12 | 45     |
| 18 | 60     |
| 24 | 60     |
| 36 | 90     |

##### 2. Sample Handling Procedures

The filter disks were kept covered in their dispenser box until they were inserted into the air sampler. At all times they were handled with clean tweezers. After sample collection, they were inserted into polyethylene envelopes which were tested to insure that they were free of antimony and barium contamination. These bags were labeled with the run number of the air sample. The purpose of these techniques was to insure that the filter disk was not contaminated by any interference trace elements, especially antimony and barium.

##### 3. Sample Preparation Procedures

For use in neutron activation analysis, the sample must be prepared in a homogeneous form. The sample geometry chosen for this work was that of a thin pellet.

Interpretation of related samples required that this geometry be reproduced for each sample. It was found that a Gelman GA type membrane filter can be inserted into 1/2 inch diameter polyethylene vials and dissolved in acetone. Upon subsequent evaporation of the acetone, the filter medium comes out of solution and forms a film at the bottom of the vial. The dust and gunshot residue remain imbedded in this film, which is readily separated from the polyethylene and easily compressed into a uniform 1/2 inch diameter pellet. These pellet samples fit well into the 1/2 inch diameter polyethylene irradiation containers and provide a reproducible geometry for subsequent irradiation counting.

In the case of the paper medium from the electrostatic precipitator, no chemical reduction has yet been developed. The filter is rolled into a cylinder of reproducible size which fits into a standard 10 dram poly-

#### 4. Neutron Activation Analysis Procedures

Neutron activation analysis is an extremely sensitive analytical technique in which the sample to be analyzed is irradiated in an intense neutron flux. Some of the atoms of the sample are converted into radioisotopes. By measuring the resulting type and energy of the decay radiation and its half life, the isotopes can be identified. By comparing the intensity of the radiation with a standard, the amount of the elemental constituent can be determined.

The gamma rays of interest in analyzing the air filter samples for antimony are the 60.7 kev gamma ray of  $^{122}\text{Sb}$  (half life 4.2 minutes) and the 564.0 kev gamma ray of  $^{122}\text{Sb}$  (half life 2.8 days). The barium radioisotope employed in this work is  $^{139}\text{Ba}$  (half life 89 minutes) having a 166 kev gamma ray. Examples of the graphical representations of these gamma ray spectra are shown in Figures 2 through 4.

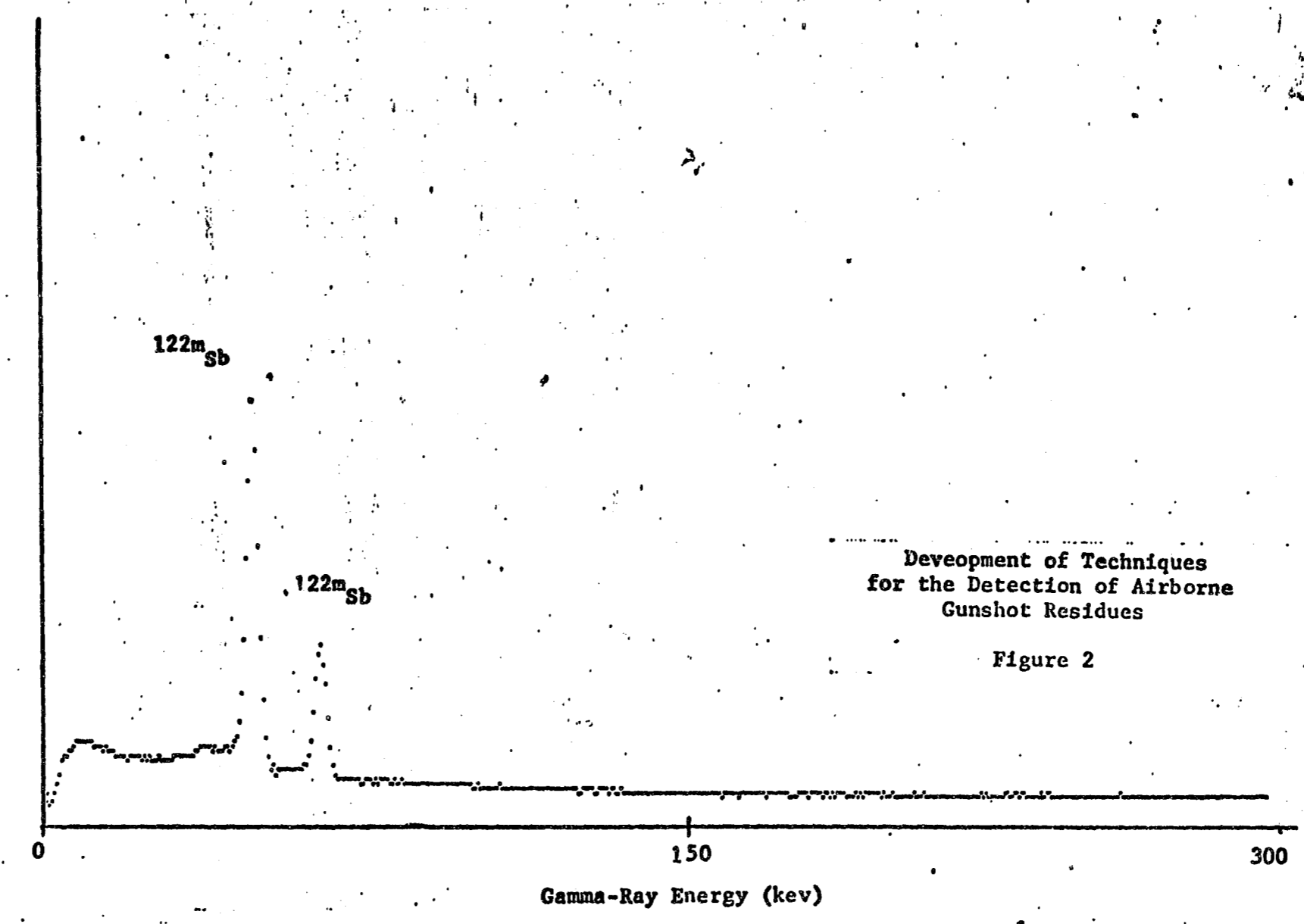
It was found that the thin Ge(Li) detector is the best one to be employed in the detection of antimony, since  $^{122}\text{Sb}$  decays emitting low energy gamma rays. The larger Ge(Li) detector is better for the detection of the higher energy photons of  $^{122}\text{Sb}$ . The gamma ray emitted by  $^{139}\text{Ba}$  is of intermediate energy, and thus can be readily detected by either detection system.

A procedure involving a 5-minute pneumatic rabbit irradiation in a thermal flux of  $2.4 \times 10^{12}$  neutrons/sec-cm<sup>2</sup> followed by a 5-minute decay time and an 800 second count time was found suitable for analyzing for the antimony and barium collected up to 12 hours after the firing test.

A procedure involving a 7-hour irradiation at the reactor core face, in a thermal flux of  $1.3 \times 10^{13}$  neutrons/sec-cm<sup>2</sup>, followed by at least a 48 hour decay period with at least a 2,000 second counting period was employed for the analysis of antimony samples collected up to 36 hours after the pistol firing.

The analysis of barium samples collected up to 36 hours after the firing involves a 5-minute pneumatic rabbit irradiation in a thermal flux of  $2.4 \times 10^{12}$  neutrons/sec-cm<sup>2</sup>. A decay of at least 60 minutes is followed by at least a 2000 second counting time.

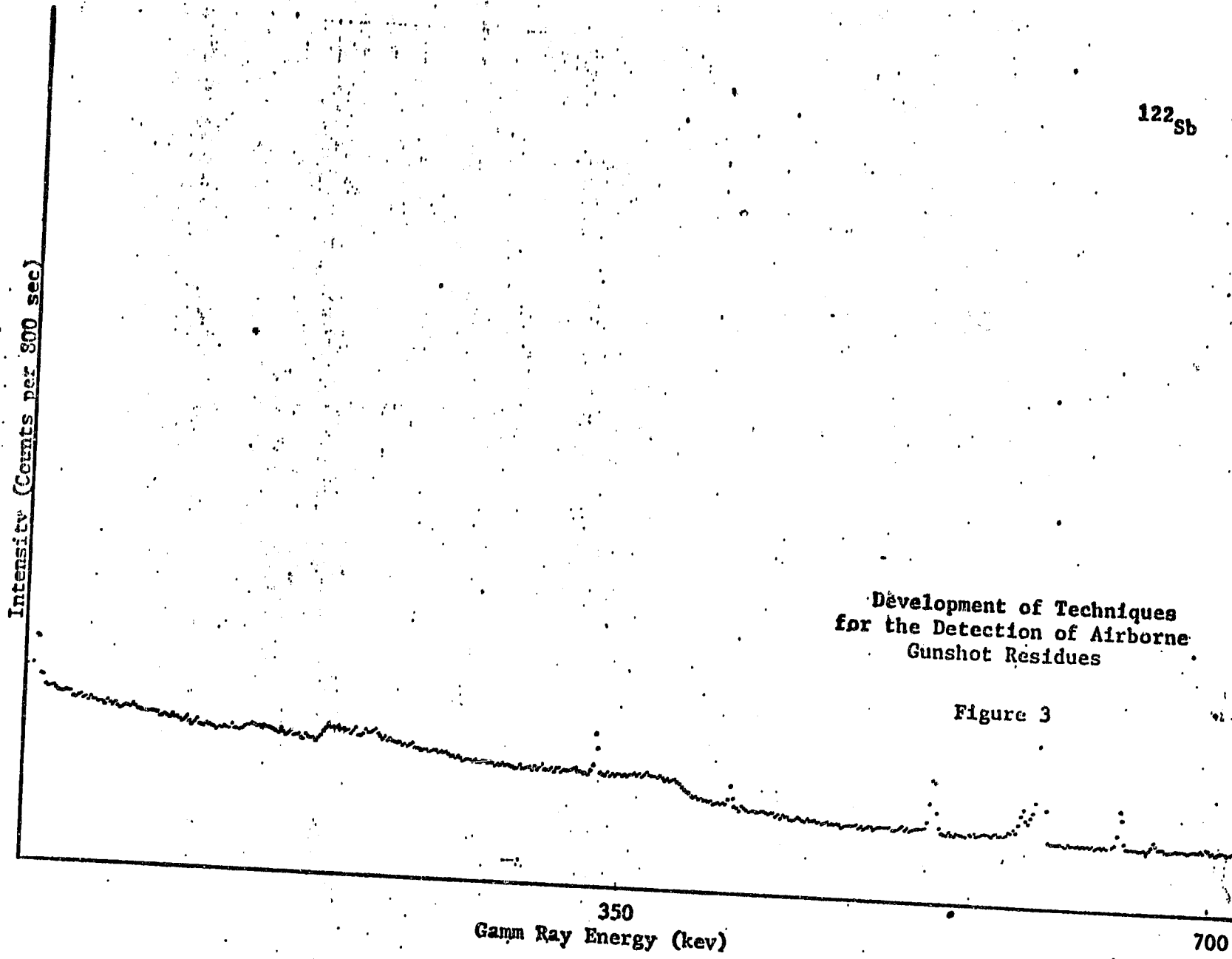
Intensity (counts per 200 sec)



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Figure 2





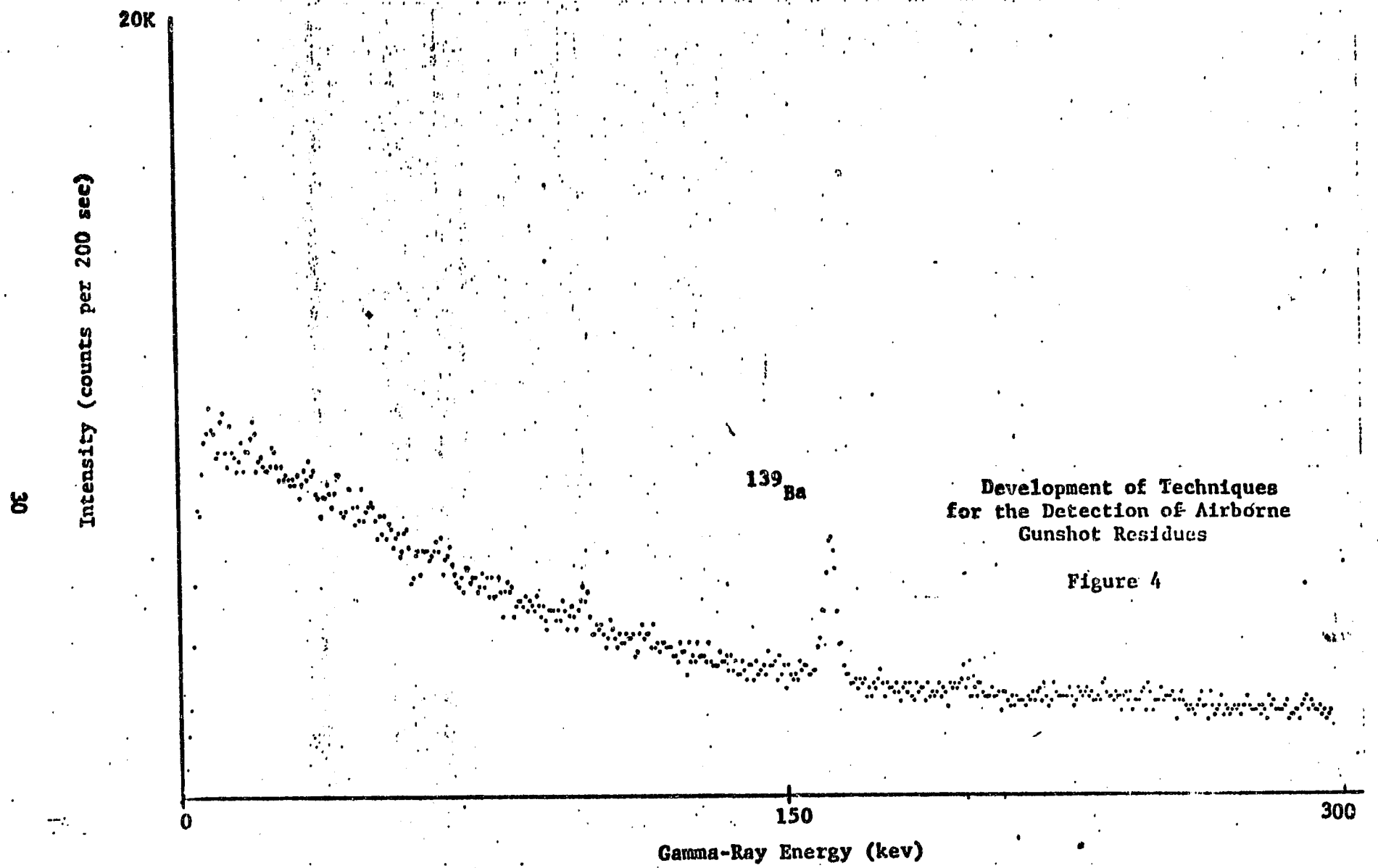
122<sub>Sb</sub>

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Figure 3

350  
Gamma Ray Energy (kev)

700



## 5. Data Processing

The raw spectral data is put on paper tape by the multi-channel analyzer. This data is then transferred to magnetic tape for permanent storage and computer manipulation. The computer programs "Energy" and "PTT" then reduce the data to give an output consisting of:

- a. peak energy, which enables the identification of the isotope
- b. peak area, a measure of the amount of the isotope present.

### Meetings with Evaluation Personnel

Throughout the first year of this project, a series of five meetings was held with evaluation personnel to obtain expert advice and criticisms on the test program as it developed.

The following persons acted in this capacity:

Captain L. D. Jenkins  
Director  
Pennsylvania State Police Crime Laboratory

Sergeant James Daffley  
Head, Ballistics Section  
Pennsylvania State Police Crime Laboratory

Dr. H. L. Guttenplan  
Professor of Law Enforcement and Correction  
The Pennsylvania State University

Dr. W. J. Moroz  
Director, Center for Air Environment Studies  
The Pennsylvania State University

Mr. Jerry Anderson  
Technician, Center for Air Environment Studies  
The Pennsylvania State University

A detailed account of the meetings is given in Appendix B.

## Findings

### A. Tests Using the Standard Collection System

Of the 14 tests conducted, a total of eight 22 caliber firings were made using the established standard sampling system consisting of the Fisher Vacuum Pump and a 2 inch Gelman GA-6 (.45 micron) membrane filter. This arrangement was the basis of comparison for all other sampling systems. The antimony and barium concentrations found as a function of time are given in Table 2. These results are plotted with respect to time in Figures 5 and 6. In addition, one 32 caliber firing was made, and the results plotted with respect to time on Figures 7 and 8.

The following observations are made concerning this data.

1. Antimony and barium are detected up to 36 hours after a single 22 caliber firing.
2. There is a great deal of scatter in the data, even between those runs which were conducted under essentially identical conditions, i.e., runs 818 and 819 and runs 820 and 821.
3. In most tests there seems to be a slow drop off of the antimony and barium collected during the first four hours followed by an exponential drop-off in the concentrations of these elements with time.
4. The antimony drops off more rapidly than the barium in both the 32 and the 22 caliber bullet test firings.
5. The 32 caliber bullet left more than an order of magnitude greater concentration of antimony and barium in the air than did the 22 caliber bullet.

Table 2

Amounts of Antimony and Barium Collected on Filter Papers\*  
 (All Data Normalized to Micrograms per 15 Minutes of Sampling Time for  
 the Firing of One Bullet)

33

| Test Number | Air Sampler and Bullet  |                        | Pretest Samples |              | Post Test Samples         |      |      |     |      |      |    |      |      |    | Exhaust Time (hrs) |
|-------------|-------------------------|------------------------|-----------------|--------------|---------------------------|------|------|-----|------|------|----|------|------|----|--------------------|
|             | Filter Medium           | Caliber                | Inside Room     | Outside Room | Time After Firing (hours) |      |      |     |      |      |    |      |      |    |                    |
|             |                         |                        |                 |              | 0                         | 2    | 4    | 6   | 8    | 12   | 18 | 24   | 30   | 36 |                    |
| 810         | Hurricane               | 22                     |                 |              |                           |      |      |     |      |      |    |      |      |    |                    |
|             | Sampling time (minutes) |                        | 30              | 30           | 15                        | 15   | 15   | 15  | -    | -    | -  | 30   | -    | -  | ~ 3                |
|             | Antimony                |                        | ND              | ND           | 0.42                      | IA   | IA   | IA  |      |      |    | IA   | -    | -  |                    |
|             | Barium                  |                        | ND              | ND           | IA                        | IA   | IA   | IA  |      |      |    | IA   | -    | -  |                    |
| 811         | Hurricane               | 22                     |                 |              |                           |      |      |     |      |      |    |      |      |    |                    |
|             | Sampling time (minutes) |                        | 30              | 30           | 15                        | 15   | -    | 195 | -    | -    | -  | -    | -    | -  | ~12                |
|             | Antimony                |                        | ND              | ND           | IA                        | 0.17 | -    | SU  |      |      |    |      |      |    |                    |
|             | Barium                  |                        | ND              | ND           | IA                        | IA   | -    | SU  |      |      |    |      |      |    |                    |
| 812         | Hurricane               | 22                     |                 |              |                           |      |      |     |      |      |    |      |      |    |                    |
|             | Fisher and GA-6         | Sampling time          | 15              | 15           | 15                        | 15   | 15   | -   | -    | -    | -  | 15   | -    | -  | ~24                |
|             | Antimony                |                        | ND              | 1.68         | 0.69                      | 0.36 | 0.14 | -   | -    | -    | -  | ND   | -    | -  |                    |
|             | Barium                  |                        | ND              | ND           | 1.03                      | 0.95 | 0.26 | -   | -    | -    | -  | ND   | -    | -  |                    |
| 813         | Fisher and GA-6         | 22 (Sampling time min) | 15              | 15           | 15                        | -    | 15   | -   | 30   | 45   | 60 | 60   | 90   | -  | ~24                |
|             | Antimony                |                        | ND              | ND           | 6.60                      | -    | 0.47 | -   | 0.18 | .084 | SU | .007 | .003 | -  |                    |
|             | Barium                  |                        | ND              | ND           | 0.67                      | -    | 0.70 | -   | 0.25 | 0.29 | SU | ND   | ND   | -  |                    |
| 814         | Gelman and GA-6         | 22 (Sampling time min) | 15              | 15           | 38                        | -    | 15   | -   | 30   | 45   | -  | 60   | 90   | -  | ~48                |
|             | Antimony                |                        | ND              | ND           | ND                        | -    | ND   | -   | ND   | ND   | -  | ND   | ND   | -  |                    |
|             | Barium                  |                        | ND              | ND           | ND                        | -    | ND   | -   | ND   | ND   | -  | ND   | ND   | -  |                    |
| 815         | Fisher and GA-6         | 32 (Sampling time min) | -               | -            | 38                        | -    | 15   | -   | 30   | 45   | -  | 60   | 90   | -  | ~24                |
|             | Antimony                |                        |                 |              | 10.4                      | -    | 9.4  | -   | 0.93 | 0.33 | -  | .030 | .007 | -  |                    |
|             | Barium                  |                        |                 |              | 7.9                       | -    | 5.4  | -   | 0.90 | 0.36 | -  | ND   | ND   | -  |                    |



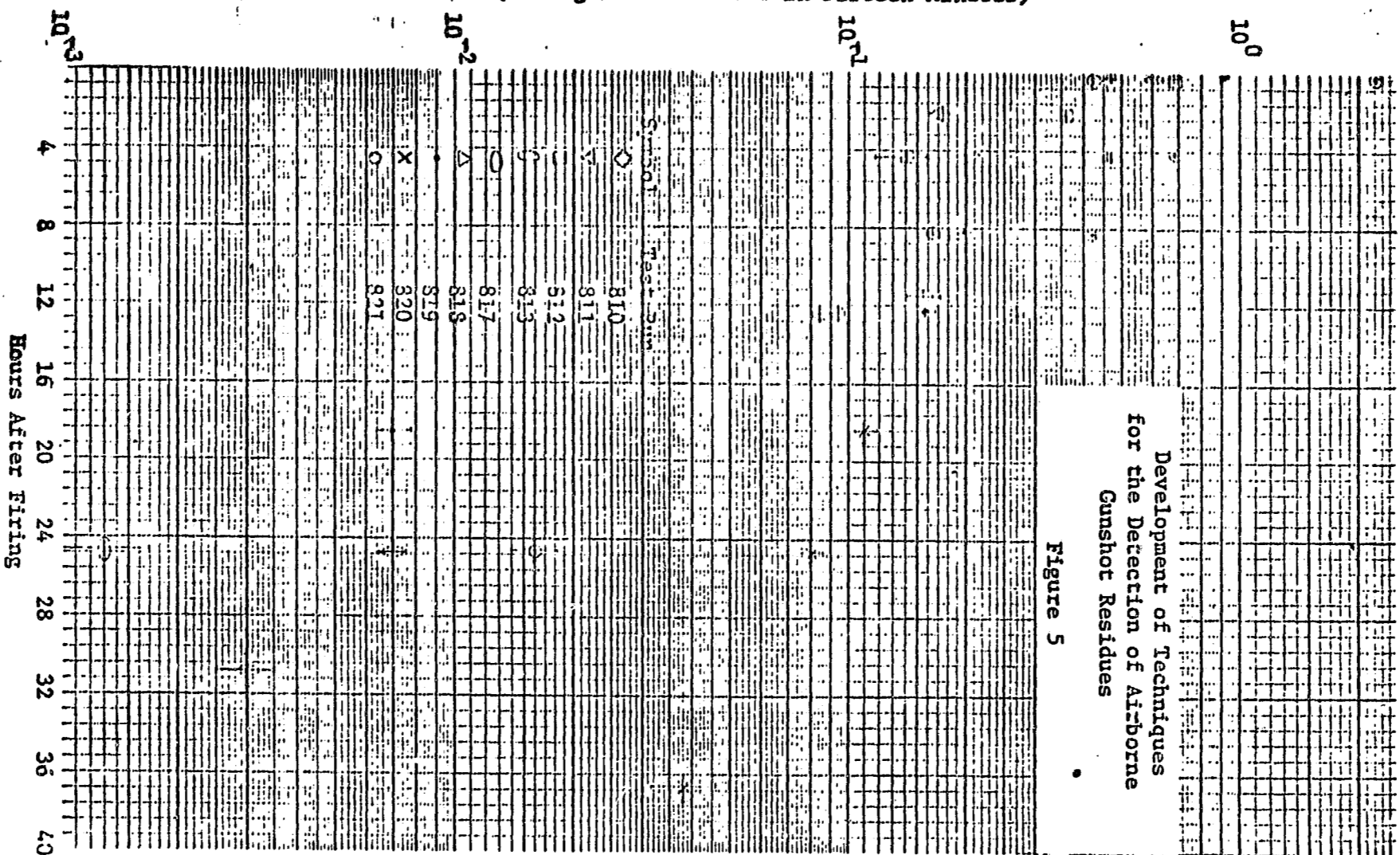
Table 2 (continued)

| Test Number | Air Sampler and Filter         |   | Pretest Samples |                | Post Test Samples         |             |                |             |                |                |                |                |             |                | Exhaust Time (hrs) |
|-------------|--------------------------------|---|-----------------|----------------|---------------------------|-------------|----------------|-------------|----------------|----------------|----------------|----------------|-------------|----------------|--------------------|
|             | Medium                         | Bullet Caliber                            | Inside Room     | Outside Room   | Time After Firing (hours) |             |                |             |                |                |                |                |             |                |                    |
|             |                                |   |                 |                | 0                         | 2           | 4              | 6           | 8              | 12             | 18             | 24             | 30          | 36             |                    |
| 823         | Fisher & 0.1 micron Nucleopore | 22<br>Sampling time<br>Antimony<br>Barium | 90<br>ND<br>ND  | 90<br>ND<br>ND | 15<br>ND<br>ND            | -<br>-<br>- | 15<br>ND<br>ND | -<br>-<br>- | 30<br>ND<br>ND | 45<br>ND<br>ND | 60<br>ND<br>ND | 60<br>ND<br>ND | -<br>-<br>- | 90<br>ND<br>ND | ~24                |
| 824         | Fisher & 0.1 micron Nucleopore | 22<br>Sampling time<br>Antimony<br>Barium | 90<br>ND<br>ND  | 90<br>ND<br>ND | 15<br>ND<br>ND            | -<br>-<br>- | 15<br>ND<br>ND | -<br>-<br>- | 30<br>ND<br>ND | 45<br>ND<br>ND | 60<br>ND<br>ND | 60<br>ND<br>ND | -<br>-<br>- | 90<br>ND<br>ND | ~24                |

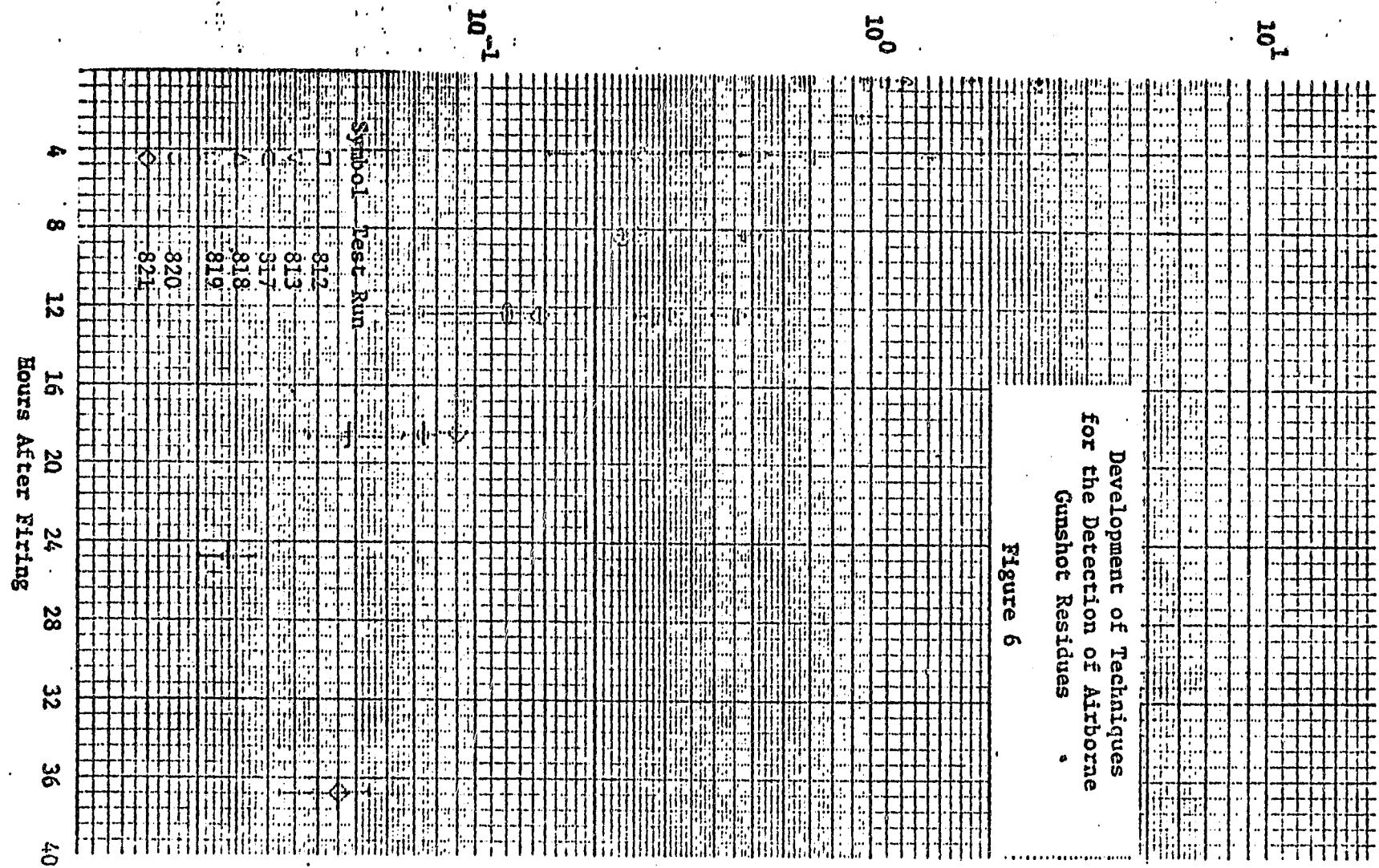
ND - Not detected  
 IA - Analysis masked by interference radioactivities  
 NI - Not yet measured  
 SU - Sample unusable because of collection or sample preparation problems

55

**Antimony Content  
(Micrograms Collected in Fifteen Minutes)**

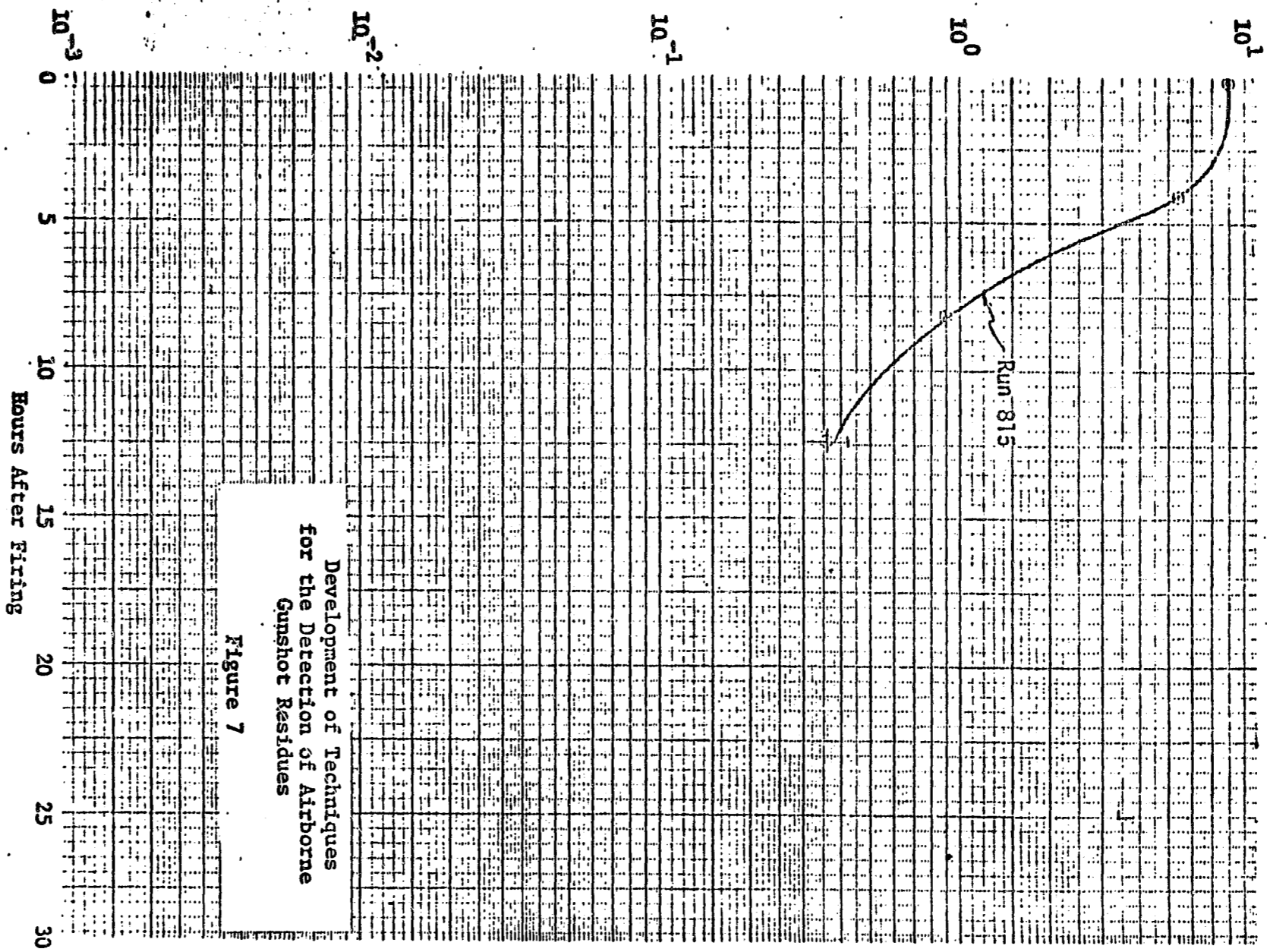


Barium Content  
(Micrograms Collected in Fifteen Minutes)



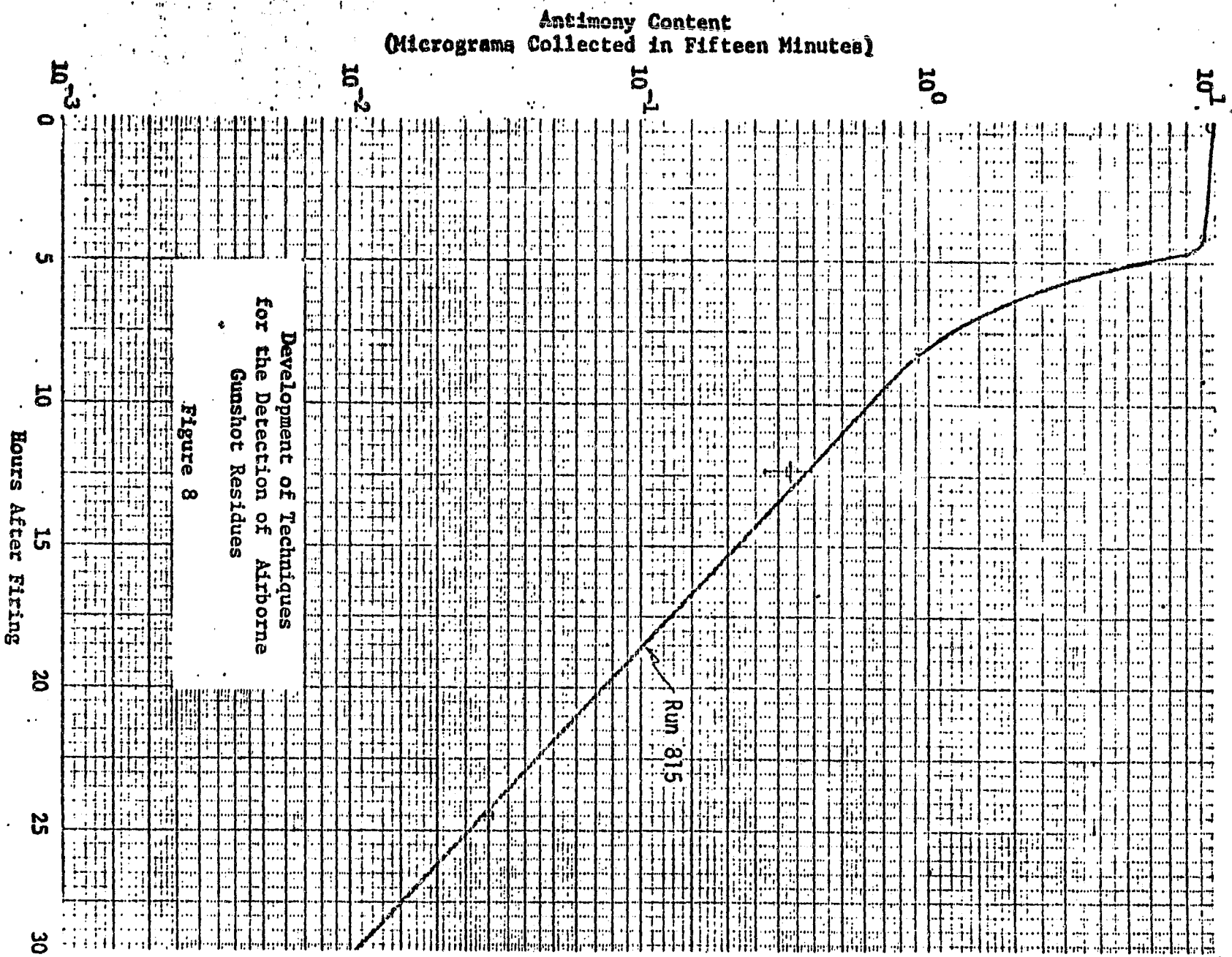


Barium Content  
(Micrograms Collected in Fifteen Minutes)



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Figure 7



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Figure 8

Run 815

6. Table 2 also reflects the intermittent elemental interferences that occurred during this testing program. All of the data points labeled IA (analysis masked by interference radioactivities) and probably most of those labeled ND (not detected) occurred because of other trace elements collected on the filter media which became more radioactive than the antimony and/or barium collected in the sample and either completely masked their activity or in some of the other cases considerably lowered the precision and therefore increased the uncertainty of the analysis.

#### B. Tests Using 0.1 Micron Filters

Two additional 22 caliber test firings were made using the Fisher Vacuum Pump and a Nucleopore filter (0.1 micron). The standard sampling times were used. As shown in Table 2, no antimony or barium was detected on the air samples despite the fact that there was no significant interferences from other trace elements. Thus despite the ability to collect smaller particles, the greatly reduced throughput made filter media having this small pore size unusable.

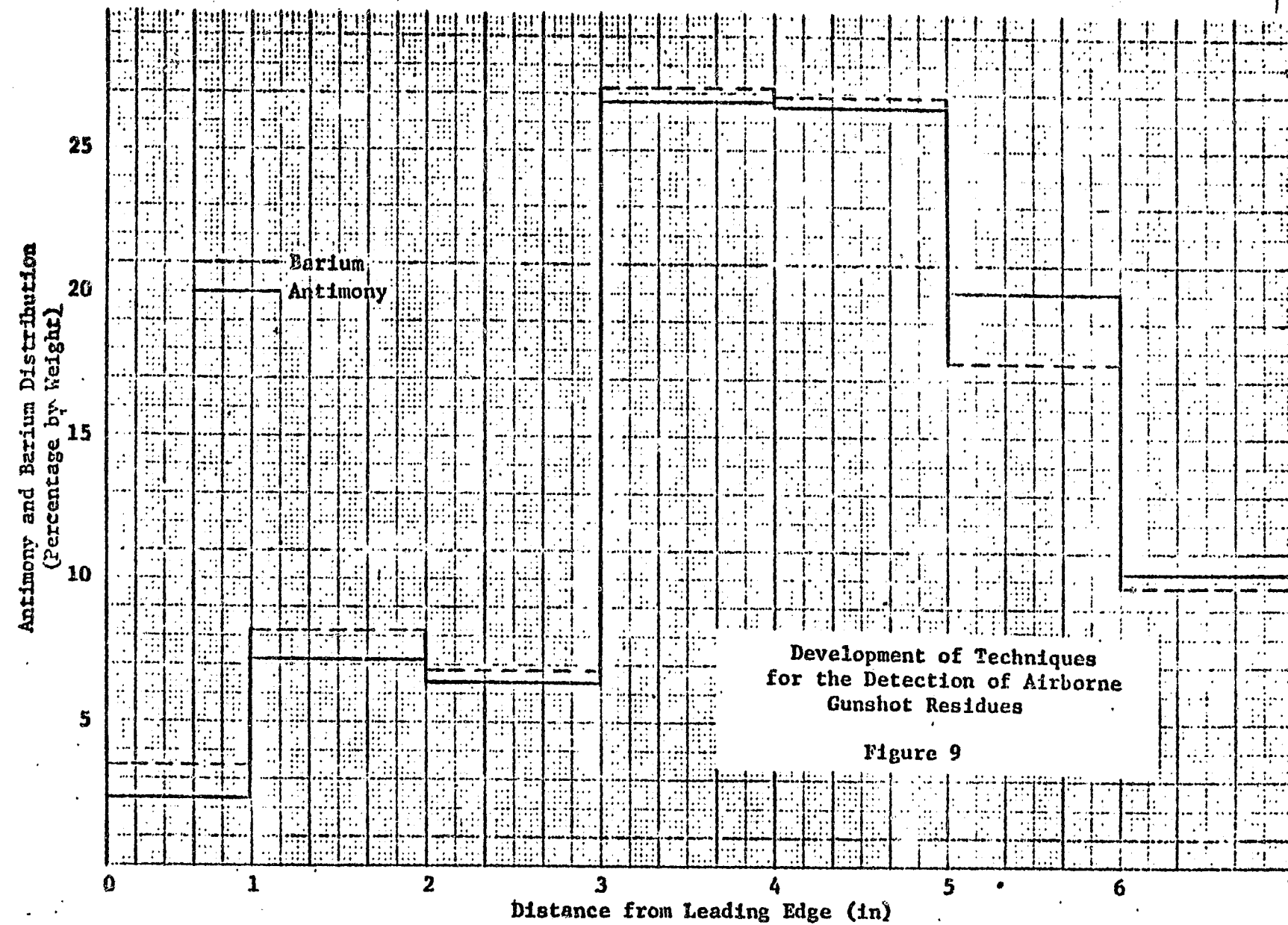
#### C. Tests Using an Electrostatic Precipitator

The test of the electrostatic precipitator involved firing three bullets in succession. This test is also summarized in Table 2. The antimony and barium concentrations were also analyzed longitudinally along the sampling tube. The seven inch long paper collection medium was cut into seven one-inch strips perpendicular to the air flow path. The concentration distribution of antimony and barium is given in Table 3 and Figure 9. Table 3 also contains the barium/antimony ratio distribution along the sample. Table 4 shows the percent by weight of the antimony and barium along the filter paper.

Table 3  
Weight Distribution of Antimony and Barium  
Along the Filter Paper\*

| Distance from<br>Leading Edge (in) | Antimony<br>(mg/15 min sample) | Barium<br>(mg/15 min sample) | Barium-Antimony Ratio |
|------------------------------------|--------------------------------|------------------------------|-----------------------|
| 0-1                                | .075 ± .008                    | .311 ± .05                   | 4.15 ± .2             |
| 1-2                                | .230 ± .008                    | .733 ± .04                   | 3.19 ± .06            |
| 2-3                                | .205 ± .008                    | .613 ± .036                  | 2.99 ± .07            |
| 3-4                                | .863 ± .015                    | 2.420 ± .05                  | 2.81 ± .06            |
| 4-5                                | .856 ± .008                    | 2.390 ± .07                  | 2.79 ± .02            |
| 5-6                                | .666 ± .012                    | 1.566 ± .06                  | 2.35 ± .04            |
| 6-7-1/4                            | .334 ± .01                     | .873 ± .04                   | 2.62 ± .06            |
| TOTAL                              | 3.23                           | 8.90                         |                       |

\* All data normalized to the firing of a 22 caliber bullet.



Antimony and Barium Distribution  
(Percentage by Weight)

Barium  
Antimony

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Figure 9

An analysis of the interference elements present in the sample was made in the same manner as with the antimony and barium. The relative concentration distribution of the four major interferences, sodium, chlorine, aluminum, and manganese, are listed in Table 5 and plotted in Figures 10 and 11.

The following observations are made concerning this data.

1. The antimony and barium distributions tend to follow the same pattern, but the barium appears to contain somewhat larger amounts of smaller particles.

2. About 53% of the antimony and 54% of the barium collected lies in a two-inch strip representing only 28% of the collection paper.

3. The sodium and chlorine distributions tend to follow the same pattern, but are considerably different from those of the antimony or barium. The sodium and chlorine are found in particles which appear to be either much smaller than the gunshot residues or somewhat larger on the average than these residues. These elements probably represent the distribution of organic dust such as pollen, lint, etc.

4. The manganese and aluminum distributions tend to follow the same pattern, but are considerably different from the two previously mentioned distribution patterns. These have a more uniform distribution across the collection paper. These elements probably represent the distribution of inorganic dust such as meteoric dust and inorganic surface materials.

Table 4

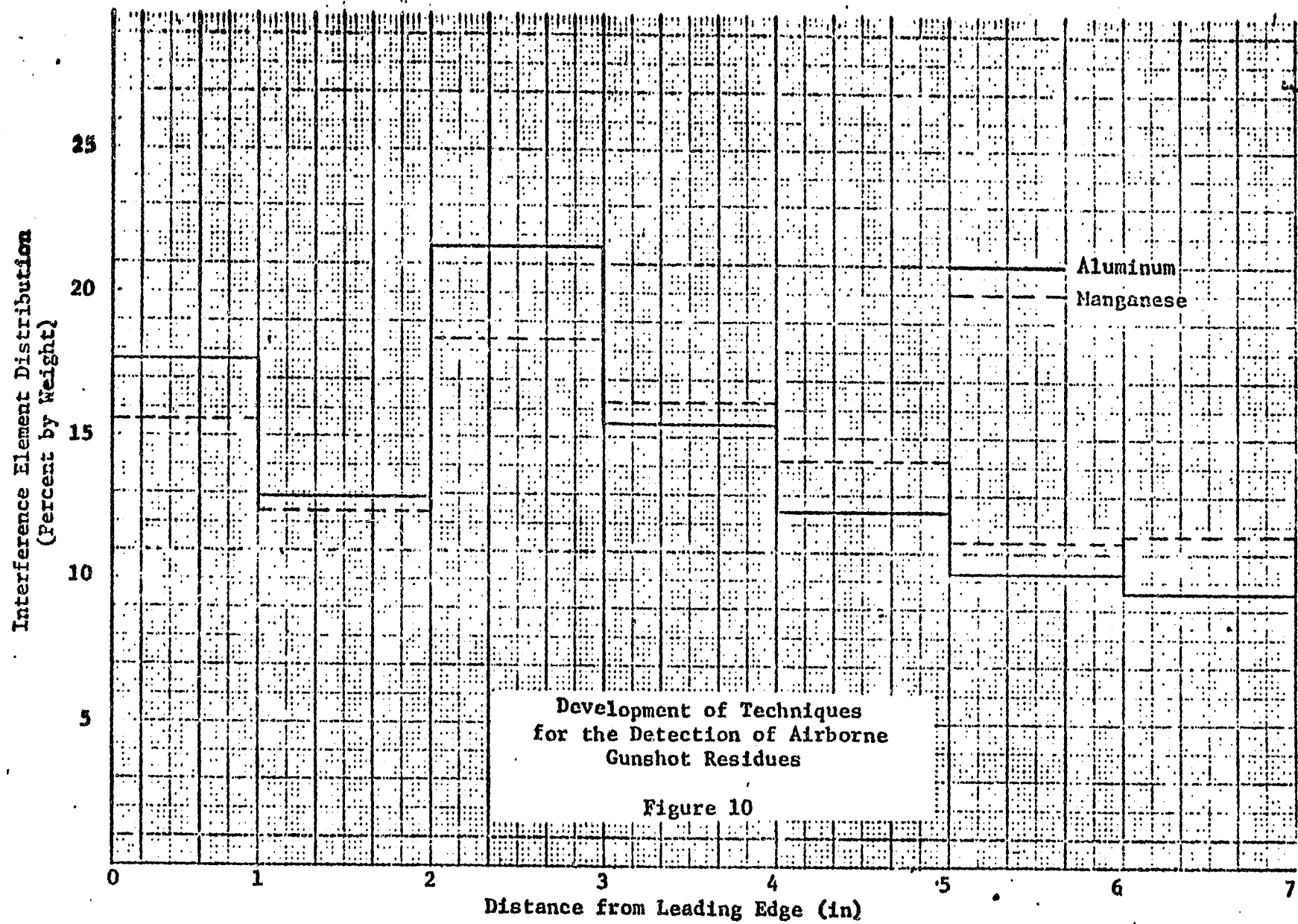
Percent by Weight of Antimony and Barium Along the Filter Paper

| Distance from Leading Edge | Sb          | Ba         |
|----------------------------|-------------|------------|
| 0-1                        | 2.3         | 3.5        |
| 1-2                        | 7.1         | 8.2        |
| 2-3                        | 6.4         | 6.9        |
| 3-4                        | 26.7        | 27.2       |
| 4-5                        | 26.5        | 26.8       |
| 5-6                        | 20.6        | 17.6       |
| 6-7-1/4                    | <u>10.4</u> | <u>9.8</u> |
|                            | 100         | 100        |

Table 5

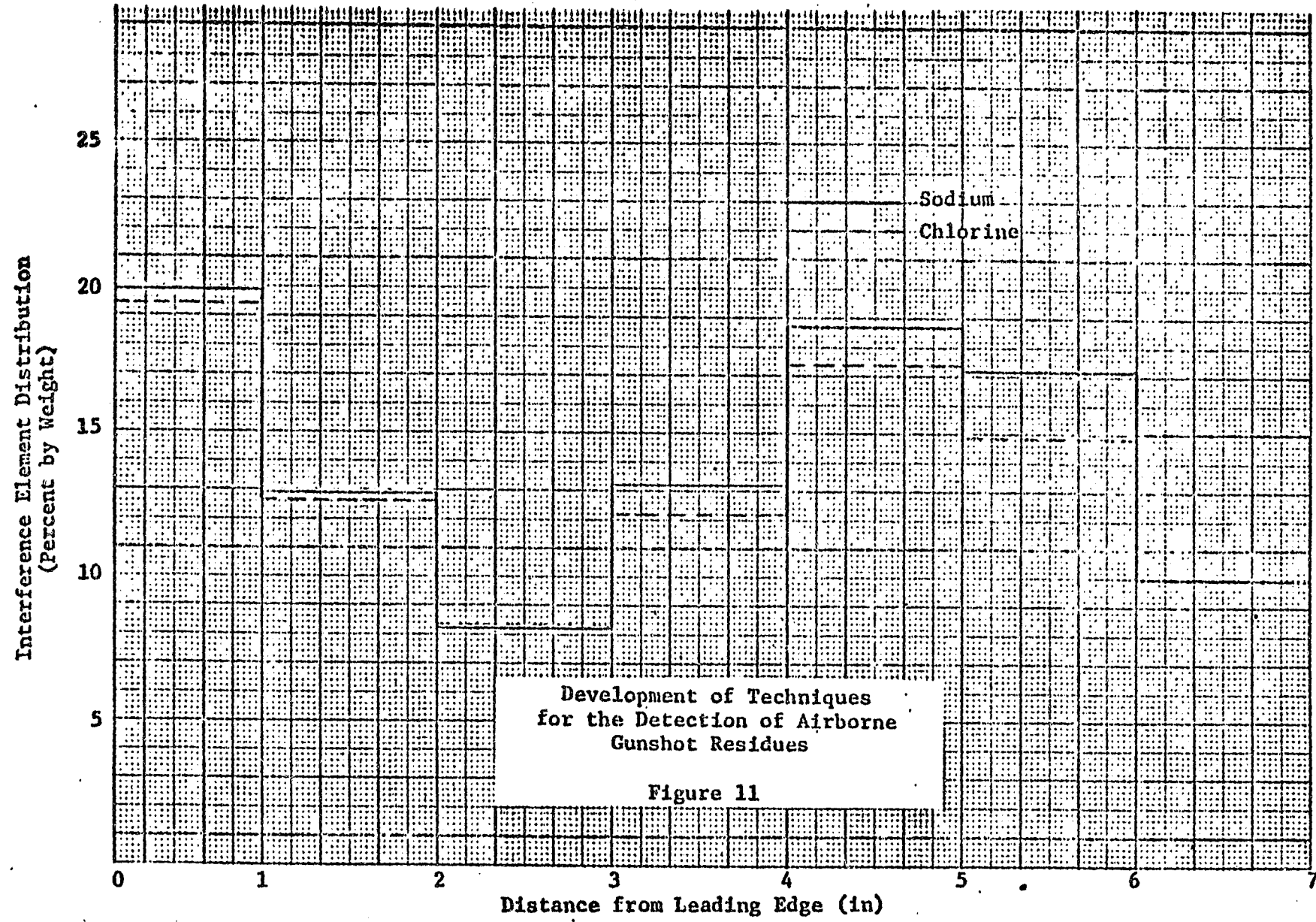
Percent by Weight of Interference Elements  
Along the Filter Paper

| Distance from<br>Leading Edge | Al   | Na   | Mn   | Cl   |
|-------------------------------|------|------|------|------|
| 0-1                           | 17.7 | 19.9 | 15.6 | 19.4 |
| 1-2                           | 12.9 | 12.9 | 12.4 | 12.7 |
| 2-3                           | 21.6 | 8.2  | 18.4 | 8.3  |
| 3-4                           | 15.5 | 13.2 | 16.3 | 12.2 |
| 4-5                           | 12.4 | 18.7 | 14.2 | 17.4 |
| 5-6                           | 10.3 | 17.2 | 11.4 | 14.9 |
| 6-7-1/4                       | 9.6  | 9.9  | 11.7 | 15.1 |



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Figure 10





5. Within the 28% range in which about 53% of the antimony and barium is deposited was found the following amounts of the interference elements:

- 32% sodium
- 29% chlorine
- 28% aluminum
- 30% manganese

D. Details of Test Runs

A detailed description of all test runs mentioned in Section "A" through "C" can be found in Appendix C.

E. Neutron Activation Analysis of 22 Caliber Bullet Components

Neutron activation analysis was employed to detect the source of antimony and barium in the Western T-22 ammunition. An unfired cartridge was carefully disassembled, and separated into four parts, namely the bullet, the gun powder, shell, and the primer. It was found that the barium occurred only in the primer while the antimony was found primarily in the lead bullet and to a much lesser extent in the primer. The barium to antimony ratio in the primer was found to be 65 to 1, a much higher ratio than is found in the collected airborne gunshot residue samples. The average barium to antimony ratio collected using the air filters is about 1.6 to 1 while that from the electrostatic precipitator run was about 2.8 to 1. Thus a considerable amount of the antimony being collected must have originated from antimony in the lead bullet.

Conclusions

A. Standard Test Results

The most important conclusion that can be made from the results of the first eleven 22 caliber firing tests is the definite confirmation that under the test conditions, airborne gunshot residues can be detected up to 36 hours after the firing of a single bullet. The 36 hour period may not be a limit as no samples were collected beyond this time span.

The ability to detect these small amounts of residue was subject to intermittent interferences from trace elements from the collection of other nonrelated airborne residues. These interferences seem especially prevalent after the settled dust in the test room had been disturbed, either by entry of test personnel prior to a test or by the exhaust from the air sampler itself. Such interferences would be a serious limitation of this technique when used in actual cases, since the lack of detection of antimony and barium would not necessarily prove that the weapon had not been fired in the sampled area. There are two potential solutions to this problem. One approach is to use an air sampler which would be selective for the particular narrow range of particle size associated with airborne gunshot residue, hence the work with the electrostatic precipitator. The second approach would be the use of post-irradiation radiochemical separation of the antimony and barium. This second approach will be held in abeyance since it would add further complications and expense to the technique.

The non-reproducibility of the data with respect to time from the eleven tests prevents as yet an accurate determination of time of firing. With the exception of a general downward trend with time, there are no constant characteristics, such as amount collected or slope of the curve, which can be related to time after firing. This may be due to inherent non-reproducibility in repeated gunshot firings despite the efforts to obtain reproducibility. The amount of primer combustion and bullet slug vaporization will definitely affect the amount of gunshot residue initially ejected into the air of the test room. Such factors as the number of airborne particles, their size distribution, and other factors such as the humidity in the room could affect the rate at which they settle out of the air.

The data spread from one test to another increased considerably with samples taken at longer times after firing. There are several analytical factors which may have contributed to this increased imprecision. One factor is the statistical uncertainty resulting from lower amounts of antimony and barium in the room. In addition, the longer irradiation times required for activating the small amounts of antimony made the sample pellet extremely fragile. Some of the pellets were found to be shattered into a coarse powder after the seven hour irradiation, making them more difficult to handle without losses and making it more difficult to obtain the standard counting geometry. Methods of post-irradiation sample dissolution will be investigated in an effort to overcome this problem.

Sample preparation problems may have been occasionally responsible for the data variations, but these did not occur very often and would not account for the variations observed.

#### B. Test Results from the 32 Caliber Revolver Firing

The single test from the firing of the H & R 32 caliber revolver followed by the standard sampling and analysis techniques shows that the problems involved in detection of single shots of larger caliber bullets are considerably less than those involved in detecting the residues from the 22 caliber bullet firings. In comparing Figures 5, 6, 7, and 8 it can be seen that the 32 caliber bullet firing left more than an order of magnitude greater concentration of antimony and barium in the air than the standard 22 caliber test.

#### C. Preliminary Test Results with the Electrostatic Precipitator

The preliminary tests with the electrostatic precipitator were very promising in several important areas of interest as given below and may help improve the reproducibility of the technique:

1. Sample collection compatibility with neutron activation analysis

The use of Whatman #50 as a sample collection media allows a significant reduction in neutron activation background interference as compared with the Gelman #GA-6 used in the standard test. This results from the lower "as received" trace element content of this paper and also from the fact that the paper can be acid washed to significantly lower this trace element content. This reduction will allow greater sensitivity in the detection of antimony and barium.

## 2. Neutron activation interferences from environmental dust

Over 53% of the antimony and barium was found to be collected in the 3 to 5 inch horizontal segment of the collection media. In this same range, only about 30% of the sodium, chlorine, aluminum, and manganese was collected. Since these four elements represent the major interference elements found to date in atmospheric dust, the analyzing of this selective portion of the sample would result in an effective reduction of about 72% in environmental dust interference.

## 3. Increased airborne gunshot residue detectibility

The amount of antimony and barium collected using the electrostatic precipitator was much larger than that collected using the standard system. Normalizing the number of shots fired and the collection time, it was found that values of 4.6 times greater in the amount of antimony and 10 times greater in the amount of barium were collected. This increase in collection efficiency is probably due to the increased sampling rate and the increased efficiency in collecting the smaller particles. This improved collection efficiency both increases the sensitivity of the method, especially for barium, and also allows the possibility of a shorter collection time.

## 4. Possible time of firing determinations

The improved collection efficiency and reduced interferences should reduce the analytical uncertainties and may provide better concentration versus time after firing data. This may improve chances of using this technique for obtaining time of firing data.

A second possibility also exists. Agglomeration is a major method by which the fine gunshot residue particles are lost from still air. In this process, many smaller particles combine into fewer large particles which eventually settle out of the air. Thus as the time after firing extends, the average size of the gunshot residue particles should increase and the peak of the antimony and/or barium distribution curve shown in Figure 9 should shift to the right reflecting this agglomeration. Thus this shift may be useful in obtaining time of firing information.

## D. Relative Size of Antimony and Barium Particles and Their Probable Sources

An examination of the barium/antimony ratio from the bullet primer shows a ratio of 65. At the same time, a gunshot residue barium/antimony ratio of 1.6 is obtained from the standard collection system data and 2.8 from the electrostatic precipitator data. This points to the appreciable antimony contribution from the bullet slug. Further comparison of this ratio for the two collection systems shows that significantly more barium is collected by the electrostatic precipitator. Also the data on the barium/antimony ratio with respect to sample position in the electrostatic precipitator (Table 3) shows higher ratios toward the leading edge of the collection paper. This data indicates that the particles formed from the lead containing the antimony are probably larger on the average than the products of primer combustion.

#### E. New Electrostatic Precipitator for Future Testing

The extension of these results into further testing is anticipated with the ordering from Del Electronics Corporation of an electrostatic precipitator, Model #ESF-100A. This unit is being specially constructed with variable electrode potential and air flow rate to allow varying the location of particle deposition. A cyclone pre-collector will be employed to significantly reduce the number of particles larger than about five microns that would enter the collection region. The aim here is to attempt to further reduce the collection of environmental contaminants. These tests will involve only one sampling period per test, as the efficiency and sampling rate are such that one sampling seriously disturbs the amount of residue suspended in the air.

#### Appendix A

#### FIRING PROCEDURES FOR THE AIRBORNE GUNSHOT RESIDUES PROJECT

by

W. Renfro and W. Jester

#### A. Pistol and Ammunition Storage

1. When not under direct supervision of Dr. W. A. Jester or Dr. W. F. Witzig, H & R 22 caliber nine shot revolver or its cylinder will be stored in a locked case in the locked radioisotope cabinet in the Control Room of the Penn State Breazeale Nuclear Reactor.
2. The ammunition will be stored in its own locked case in the locked radioisotope cabinet in Room 4 of the Breazeale Nuclear Reactor.

#### B. Prefiring Steps

1. Since only Dr. Jester and Dr. Witzig will have keys to the two cases, one of them must be present to begin the prefiring procedure. In addition, only senior reactor operators have the keys which will allow the opening of the locked radioisotope cabinet in the Control Room.
2. The gun case and the required amount of ammunition will be signed out and transported to the test area just prior to testing, with Dr. W. F. Witzig or Dr. W. A. Jester in attendance.
3. The unloaded pistol or the pistol cylinder will be removed from the gun case and mounted in the firing apparatus.
4. The proper aiming of the pistol will be visually confirmed.

5. The correct number of shells will be inserted into a holder in the glove box and the box sealed. At no time after the shells are placed in the glove box will anyone be allowed in the polyethylene room until the cylinder has been removed from the revolver.

C. Firing Steps (a minimum of two persons from a group consisting of W. Renfro, Dr. W. Jester or Dr. W. Witzig will be present at all times that the pistol is not locked in its case).

1. An announcement will be made over the reactor public address system stating that all unauthorized persons must remain clear of the testing area.
2. Signs to this effect will be placed at all possible entrances to the testing area on rope barriers.
3. A check to see that all unauthorized persons are out of the test area will be made.
4. The experimenter will announce to those in the test area that he is about to load the pistol.
5. The correct number of shells for the test will then be loaded into the pistol using the glove ports on the glove box, and the pistol will be moved to its firing position.
6. A final check will be made to see that the testing area is clear of unauthorized persons.
7. Authorized persons will move to a position such that the glove box is between them and the bullet catcher.
8. The experimenter will announce to those in the test area that he is about to fire the pistol.
9. The pistol will be discharged by the experimenter manually pulling the trigger.

#### D. Post-firing Steps

1. Immediately after firing, the cylinder will be removed from the pistol.
2. An announcement will be made that the firing is completed and the signs removed.
3. As soon as the air sampling procedures will permit, the pistol or its cylinder will be taken from its holder and removed from the glove box by means of its access port.
4. The pistol cylinder will be checked for unfired bullets, cleaned, locked in its carrying case, and returned and signed in to its storage area.
5. If the pistol without its cylinder is to remain in the firing apparatus it also will be cleaned at the conclusion of the test.

Appendix B

MEETINGS WITH EVALUATION PERSONNEL

1. On October 21, 1970, a meeting was held at the Pennsylvania State Police Crime Laboratory, Harrisburg, Pa., between project personnel and Captain Ludwick D. Jenkins, Director of the Laboratory, and Sergeant James Deffley, Head of the Laboratory's Ballistics Section. The proposed testing facility and initial testing procedures were discussed at this time. With a few minor comments, these were enthusiastically endorsed by Captain Jenkins and Sergeant Deffley. It was at this time that Sergeant Deffley confirmed that the type of firearm to be employed in initial testing and on the construction of the bullet catcher would be satisfactory. It was also suggested that some tests should be conducted with ammunition containing only the primer, and the primer and powder, as well as with complete bullets, in order to pinpoint the sources of the suspended residues. The assistance of the Crime Laboratory was offered in preparing these types of ammunition.

Captain Jenkins stated that if this technique proves successful, he is anxious to place the required air sampling equipment in all of the State Police's mobile crime laboratories which will soon be used throughout the State of Pennsylvania to investigate major crimes. A second meeting with Captain Jenkins and Sergeant Deffley was planned at the completion of the construction of the testing facility.

2. On November 2, 1970, a meeting was held at The Pennsylvania State University's Center for Law Enforcement and Correction, University Park, Pa., between project personnel and Dr. Henry L. Guttenplan, Professor of Law Enforcement and Correction. Dr. Guttenplan also endorsed the conceptual design of the testing facility and the initial testing procedures. He stressed that a major consideration must be the prevention of sample contamination by outside sources of antimony and barium. He provided information concerning his experience with the distribution of gunshot residues resulting from the firing of revolvers and speculated on how it might affect the results of the proposed tests. He also stressed that only after sound experimental testing under controlled conditions is completed can practical field testing of the technique be initiated in order that meaningful samples and accurate interpretation of results can be obtained. A second meeting with Dr. Guttenplan was scheduled to take place at the Breazeale Nuclear Reactor after the completion of the testing facility.
3. On May 21, 1971, after completion of the test room, a Project Evaluation meeting with personnel from the State Police Crime Laboratory and Dr. Guttenplan covered the following items:
- a. Test procedures and results acquired to date were presented and discussed.
  - b. A demonstration of the 22 caliber revolver firing in the test room was conducted.

c. Comments from evaluation personnel

(i) Evaluation personnel gave their approval concerning the testing facilities and procedures being employed.

(ii) Sgt. Deffley inquired about whether or not multiple shots gave a substantial difference in the amount of residue left in the air as compared to a single shot. Subsequent testing should answer this question.

(iii) Captain Jenkins suggested that the next caliber firearm to be tested should be a 38 caliber S&W or Colt manufactured with a six-inch barrel. This would be considered to be an in-between caliber (between the 22 and 45 caliber firearms often encountered in criminal cases). This also is the firearm carried by the State Police personnel. He stated that he would inquire about the possibility of having the State Police loan such a firearm to the project for testing purposes.

(iv) Dr. Guttenplan suggested that air samples should be taken at various positions in the room since the residue concentrations may be a function of where in the room the pistol was fired. Dr. Guttenplan also suggested that all test shots be fired from the same chamber in the revolver since slight variations in chamber and cylinder dimensions may change the amount of gunshot residues ejected into the test room.

4. The May 26th meeting with Dr. William Moroz was held at the office of The Pennsylvania State University Center for Air Environment Studies. The meeting was held to obtain information from Dr. Moroz and his staff on the nature and settling rates of airborne particulates and on the type of sampling equipment and techniques that should be investigated. Dr. Moroz gave the following information.

a. In a combustion process such as a firearm discharge, the particles formed are usually in the submicron region (0.1 to 0.01 $\mu$ ). Such small particles can remain suspended in still air for periods up to three months. They are lost from the air primarily by coagulating to form large particles which then fall out under the influence of gravity.

b. The filters currently being employed in this project therefore have too large a pore size (0.45 $\mu$ ), and thus it is primarily the coagulated residue particles which are being collected with the small particles passing right through the filter.

c. The dust concentration in the room may influence the coagulation rate.

d. The process of diffusion by which the residue would be spread evenly throughout the room probably proceeds quite slowly and may produce concentration differences in the room, even up to four hours after firing.

e. A ratio of the large particles to small particles may be a relevant measure of the time after firing, since the amount of small particle coagulation should be a function of time.

f. It was suggested that for portable field sampling, a 10 liter vacuum bottle may be the answer.

5. Dr. Moroz suggested that Mr. Renfro contact Mr. Jerry Anderson, a technician at the Air Environment Center, to discuss possible air sampling equipment that could perform particle size separations. This meeting was held on May 31, 1971. Mr. Renfro explained the program and inquired whether some combination of cascade impactors and air filter collectors could be employed to accomplish this type of separation. Mr. Anderson supplied the following information:

a. A cascade impactor is not adequate for particle size separation in the submicron range. The use of a 0.1-0.01 final air filter would necessitate a pressure differential across such a filter of over 100 psi to obtain sufficient flow through the impactor to separate one micron particles on the most sensitive stage of an impactor. It would be difficult to construct an air filter system to operate at such pressure differences.

b. An electrostatic precipitator may provide the desired filtering requirements since such equipment can provide some classification by particle size in commercially available units which combine high sampling rate, portability, and moderate price. He stated that the Air Environment Center could provide a short term loan of such equipment for testing its usefulness.

c. Concerning air sampling filters, he suggested a filter material called "Nuclepore" made by the General Electric Company which offers less flow resistance for the same pore size than other filter materials such as the Gelman and Millipore membrane filter. Samples of these filters were given to Mr. Renfro to evaluate their handling properties and activation analysis interference constituents.

## Appendix C

### DESCRIPTION OF TEST RUNS

#### 1. Test Run Number 810

a. Pretest samples showed no evidence of antimony or barium inside or outside the test room prior to commencing the test firing.

b. In all post-firing samples, there was found to be a large amount of interference activity later identified as uranium-239. It is suspected that this interference came from the activation of collected particles of concrete dust produced in fastening the bullet catcher to the concrete floor. This dust was thought to be subsequently disturbed by the high velocity air currents from the Hurricane air sampler. Exhausting the room for about three hours at the end of the test eliminated this source of interference from subsequent tests.

c. There was enough antimony in the air sample obtained immediately after firing to obtain an analysis using a three hour irradiation period and a 24 hour decay period.

d. No 15 minute samples were taken during the 8 and 12 hour intervals after firing as proposed in the initial testing procedures because of the difficulty of manually taking samples throughout the night. This difficulty led to the ordering of an automatic clock timer to turn the air sampler on and off.

#### 2. Test Run Number 811

a. Pretest samples showed no evidence of antimony or barium in the air inside or outside the test room prior to the test firing.



b. Because of mistakes in the timing procedure involved in taking the 4 and 6 hour samples, this run was aborted at that point.

c. In the samples taken, no large interference peaks occurred in the energy range of the antimony and barium radioisotopes, but a large Compton continuum from higher energy gammas tended to mask the barium and antimony peaks. The major interference element in this case was sodium as sodium-24. A possible source of this sodium is the caustic soda used in the regenerating of a demineralizer located in the same space as the test room. The high velocity air currents from the Hurricane air sampler may have stirred up debris accumulated in the test room during construction.

d. The test room was exhausted overnight for about 12 hours.

### 3. Test run Number 812

a. The pretest samples taken outside of the room picked up antimony in the air but no barium, while the pretest sample within the room showed no evidence of antimony or barium. It was later determined that the demineralizer located in the same area as the test room had been regenerated the day before this test using the powdered caustic soda. It is suspected that the outside antimony contamination came from this source or from some other chemical employed in the regeneration of the ion exchange system, since in no subsequent test was either antimony or barium detected outside the test room.

b. The Hurricane air sampler broke down after taking the two pre-firing samples. Thus, a sampler employing a Fisher vacuum pump was quickly inserted into the test room to take post-firing samples.

c. An immediate effect of the change of air samplers was a reduction in the interference Compton continuum. The Fisher air sampler sampled at a slower rate and the exhaust air stream was returned to the room at the opposite end from where the sample was being taken. Thus, the problem of stirring up dust and debris from the walls and floor of the test room was lessened and usable antimony and barium data was obtained from the first three samples taken after the firing test.

d. Again no measurements were taken during the night hours. One fifteen minute sample was taken the following morning, but no evidence of antimony and barium was seen.

e. The results of this test showed the rapid fall off of the gunshot residues collected with time and thus demonstrated the need for using longer sampling time for samples taken longer than about four hours after the initial firing.

f. A one day exhaust time was employed in an effort to further reduce dust and debris which accumulated in the room during construction.

### 4. Test Run Number 813

a. At this point an electric timer was obtained to automatically control the air sampler. This allowed the taking of one sample during the night period. A modification of the post-firing testing program was instituted as given below.

| Time after firing<br>(Hours) | Air Sampling Time<br>(Minutes) |
|------------------------------|--------------------------------|
| 0                            | 15                             |
| 4                            | 15                             |
| 6                            | 30                             |
| 12                           | 45                             |
| 18                           | 60                             |
| 24                           | 60                             |
| 30                           | 90                             |

b. Pretest samples showed no measurable amounts of antimony or barium inside or outside the test room. Thus the antimony interference detected in test 812 had been removed by the venting of the area outside the test room.

c. The 18-hour sample was destroyed during the sample preparation and thus its antimony and barium content could not be determined. This particular timing interval was dropped from all subsequent measurements.

d. The samples taken at intervals longer than 12 hours after the test firing could not be successfully analyzed for antimony and barium using the short irradiation procedures because of the small amount of these elements collected even with the longer sampling times. These samples were therefore only analyzed for antimony using the procedure involving the five megawatt hour irradiation at the reactor core face.

e. Again a one day exhaust time was employed.

f. It turned out that test run 813 gave the most consistent results of any of the 22 caliber bullet firing tests taken during this quarter.

### 5. Test Run Number 814

a. The Fisher vacuum pump was not available for this test run, so a Gelman vacuum was employed. This pump had a significantly lower sampling rate, which is probably the reason why no antimony or barium was observed on any of these samples despite a low level background interference activity.

b. The 38 minute sample taken at the beginning of the firing interval resulted from a timing error.

c. The test room was exhausted over a weekend.

### 6. Test Run Number 815

a. This test involved the firing of a single 32 caliber bullet from an inexpensive H&R revolver which was made available for use in this project for a few days. Test conditions employed were the same as those used in test run 814 except that the Fisher vacuum pump was employed.

b. No pretest samples were employed because of the long exhaust time at the end of test run 814.

c. The same analysis procedures described in Section 4 above (test run 813) were employed in this test.

d. At the end of this run the test room was exhausted for about one day. To check the effectiveness of the bullet catcher, the location of the five 22 caliber slugs and the one 32 caliber slug needed to be determined. Thus, the test room was opened and the slugs removed. All six slugs were found in the range of about 10 to 15 inches into the bullet catcher. At that time the floor was cleaned using a vacuum cleaner in an attempt to remove deposited dust and other debris. The room was then resealed and exhausted for another 12 hours.

### 7. Test Run Number 816

a. This test did not relate to the current project and did not involve the discharge of a firearm. Thus the results will not be reported.

### 8. Test Run Number 817

a. This test employed the 22 caliber revolver using the sampling procedures established in run 814 and the analysis procedures established in run 813.

b. For the first time, barium was found suspended in the air of the test room in the sample taken prior to firing, but no antimony was detected. The reason that this occurred despite the 12 hour exhaust procedures after the disturbance of the interior of the test room is not known.

c. As in the case of run 811, a high interference Compton continuum occurred and masked most of the antimony and barium results on the samples taken after the initial post-firing sample. Again it appears that incomplete exhaustion of the test room after disturbing its interior contents is probably the cause of this problem.

d. The test room was exhausted for about a day at the completion of this test.

#### 9. Test Run Number 818

a. In order to insure the removal of all trace contaminants, both inside and outside the test room, 90 minute sampling periods were employed in these areas prior to testing. No antimony or barium was found on the samples.

b. It was found that the interference activities obtained during this run were significantly lower than those observed during test run 817, again indicating that entering the test room caused the problem encountered in this previous run.

c. Problems developed in preparing the eight and the 24-hour samples, and thus no results were obtained for these samples.

d. At the end of this test run, a 24-hour exhaust period was employed.

#### 10. Test Run Number 819

a. This test involved the .22 caliber revolver using the sampling procedures established in run 814 and the analysis procedures established in run 813.

b. No antimony or barium was found in the pretest samples.  
c. Sampling ended after the 12 hour sample due to collection difficulties.

d. A 24 hour exhaust period was employed at the end of this test run.

#### 11. Test Run Number 820

a. The sampling procedure was altered so that a 15 minute sample was taken immediately after firing, and the 90 minute sample previously taken at 30 hours was taken instead at 36 hours. All other sampling times were as previously established.

b. No antimony or barium was found in the pretest samples.

c. Using a new technique for barium analysis, the barium content was measured for the first time in the samples collected at 18, 24, and 36 hours after firing.

d. A 24 hour exhaust period was employed at the end of this test run.

#### 12. Test Run Number 821

a. This test was an exact duplicate of Run 820.

b. No antimony or barium was found in the pretest samples inside or outside the test run.

c. Sample interferences resulted in antimony not being detected at times after firing of 4, 18, and 24 hours and barium not being detected in the 4 and 24 hour samples.

d. A 24 hour exhaust period was employed at the end of this test run.

#### 13. Test Run Number 822

a. No pretest samples were taken in this run.

b. The test involved three successive .22 caliber firings followed by a 15 minute sample with a Bendix Electrostatic Precipitator with its most limiting orifice.

c. A 36-hour exhaust period was employed at the end of this run.

14. Test Run Number 823

a. No antimony or barium was found in pretest samples. The test procedures of Run Number 820 was used, but the filter used was a 0.1 micron Nucleopore filter. The pressure differential was increased from 8 to 20.

b. The absence of any Sb or Ba is attributed to the greatly reduced flow rate of the 0.1 micron filter.

c. A 24-hour exhaust period was employed at the end of this run.

15. Test Run Number 824

a. No antimony or barium was found in pretest samples.

b. This test was a duplicate of test run number 823 and the same results occurred: No measurable amounts of antimony or barium were collected in any of the samples.

c. A 24-hour exhaust period was employed at the end of this run.

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