

Recent Advanced Identification Method For Gunshot Residues: A Review

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ABSTRACT

Gunshot residues (GSR) can be analyzed through physical and chemical characteristics. Those residues are most commonly deposited on the back and front side of both hands, palms of both hands, clothes, and hair of a shooter. It comprises burnt and unburnt particles from the explosive primer, including lead styphnate, barium nitrate, antimony sulfide, and mercury fulminate. The residues can be collected through organic and inorganic methods after collection residues were taken for analysis chemical tests to detect the presence of nitrites, lead, copper, antimony, barium, etc. The recent advanced identification method is Scanning Electron Microscopy combined with Energy Dispersive Xray spectroscopy (SEM/EDX), Ion Beam Analysis with Particle Induced X-ray Emission (IBA - PIXE) Graphite Furnace Atomic Absorption Spectroscopy (GF - AAS), Inductively Coupled Plasma Spectrometry (ICP - MS), Laser-Induced Breakdown Mass Spectroscopy (LIBS) and Inductively Coupled Plasma Atomic Emission Spectrometry (ICP - OES). The residues on the hands, clothes, etc. can confirm who has discharged the firearm. The detection of GSR particles helps the forensic expert determine the number of shots fired and the proximity between the gun and its intended target. GSR analysis can link individuals or objects to action involving the



transfer of residue, which means "every contact leaves a trace", and comes under the principle of exchange. The information received from contact can be helpful for the investigators to identify potential perpetrators, reconstruct crime scene events, and corroborate and challenge witness testimony. The conclusion is that analysis can help the investigators to determine whether the suspected shooter may have fired a firearm or not and to estimate the range of fire from muzzle to target.

1. INTRODUCTION

Gunshot residues are also known as cartridge discharge residues or firearm discharge residues which are particles produced during discharge of a firearm. These minute portions of matter are of micron dimensions, a combination of organic and inorganic compounds, primer, propellant from the cartridge cases, bullet, or from the gun itself. GSR particles are produced from the gun and discharged to the victim, the shooter, or different objects present at the crime scene. GSR patterns that are affected by the factors include type of ammunition, firearm, barrel length, firing angle, atmospheric conditions, target material, and muzzle end to target. The identification of gunshot residues is commonly performed to determine whether the shooter has fired a gun, to estimate the distance of fire, and to determine the bullet entrance hole. When the projectile is expelled at the same moment cartridge is also discharged then residues come out. Those residues are composed of organic, and inorganic vapors, gases, and particles. These residues are expelled from the muzzle end and also from the gap between the drum and barrel rear end and ejection port. Inorganic GSR particles established from the combination of primer which has been consisted of lead styphnate, barium nitrate, and antimony sulfide used as an initiator, oxidant, and fuel respectively.[1]

GSR analysis helps investigators acquire valuable information in cases such as suicide, homicide, mass shootings, terrorism, accidental shootings, etc. The most intensive gunshot residue particles are found on the hand of the shooter and was observed that the persistence of GSR is a long time found on the barrel of the firearm in which there is less chance of affecting environmental conditions in the barrel area. Therefore, analysis of gunshot residues found on the barrel of the gun takes a long interval. The average half-time of GSR particles found on the hand of the shooter and residues on the clothes of the shooter are found to be one hour. Because of that, the time factor is most important once in collecting samples from the findings. The identification of trace elements from gunshot residues helps the investigators to get valuable information regarding the suspect and victim. The inorganic gunshot residues are obtained from the primer which is composed of Barium nitrate, antimony sulfide, and lead styphnate, and organic gunshot residues are obtained from propellant which is composed of nitrites and nitrates. When a firearm is discharged the heat and pressure lead to the deposition of organic and inorganic gunshot residues in the gunpowder and shot shell. [20]

2. COMPOSITION OF GUNSHOT RESIDUES

Gunshot residues are composed of organic and inorganic components with inorganic residues obtained from primer and organic ones from propellants. The inorganic gunshot residues consist of metallic components such as lead, barium, and antimony and organic gunshot residues consist of nitrates and nitrites, it depends on the commercial brand and types of ammunition. Other elements such as calcium, aluminum, iron, copper, zinc, nickel, silicon, and potassium can also be found. The main component of propellants is diphenylamine which is used as a stabilizer to prevent the decomposition of explosive products like nitrocellulose and nitroglycerine which are present in smokeless powders. The inorganic gunshot residues may be accidentally removed from the shooter's hand due to some environmental conditions, but the diphenylamine should remain in the shooter's hand so it indicates the presence of gunshot residues and provides useful information to the investigators for the identification of suspects in firearm-related crimes. For the identification of inorganic gunshot residues, the color tests used include the Harrison Gilroy test, Sodium Rhodizonate test, and the instrumentation techniques are Neutron Activation Analysis(NAA), Atomic Absorption Spectroscopy(AA), Scanning Electron Microscopy -Energy Dispersive Spectroscopy(SEM-EDX), Inductively Coupled Plasma -**Optical** Emission Spectroscopy(ICP-OES), Atomic Force Microscopy(AFM) and for the identification of organic gunshot residues color test include Walker test, Griess test, Dermal nitrate test, and the instrumentation include Raman Spectroscopy, Attenuated Total Reflectance – Fourier Transform Infrared Spectroscopy(ATR-FTIR), Gas Chromatography-Mass Spectroscopy(GC-MS), High-Performance Liquid Chromatography(HPLC). [4]

3. COLOUR TEST FOR IDENTIFICATION OF GUNSHOT RESIDUES

3.1 IDENTIFICATION OF INORGANIC GUNSHOT RESIDUES

3.1.1 Harrison and Gilroy test

Take a cotton swab or cotton piece and dipped it in 0.1N HCL and then apply on hand of the shooter, after that add 2-3 drops or 2-10% of triphenyl methyl arsonium iodide solution to the cotton. If -an orange ring is formed indicates the presence of antimony. After drying it for 2-3 minutes, 5% of freshly prepared Sodium Rhodizonate solution is added, if a red color appears indicating the presence of lead or barium. Kept it dry. Then add 1:20 HCL, if the red color changes to blue indicating the presence of lead, and the red color is retained confirming the presence of barium.

3.1.2 Sodium Rhodizonate test

Take a filter paper and moisten it with 0.1N HCL over the bullet hole. Keep the filter paper dry using a hot air blower. Then add sodium rhodizonate solution. Then orange color will appear indicating the presence of lead.

3.2 IDENTIFICATION OF ORGANIC GUNSHOT RESIDUES

3.2.1 Walker test

This test is utilized for the examination of nitrates in half-burned and unburnt propellants. Take a desensitized photographic paper. They put the paper in 5% of α - naphthylamine and sulphanilic acid. Then this paper is pressed against the bullet entry hole. Keep it for dry around 5 minutes. The orange spot will form. This spot indicates the presence of nitrite particles.

3.2.2 Griess test

Take the sample and add ether. Separate the sample into equal amounts in two test tubes. Then test tube 1 takes caustic soda and Griess reagent and test tube 2 takes Griess reagent alone. In test tube, 1 solution changes to pink color, and in test tube 2 changes to colorless indicating the presence of nitrates

3.2.3 Dermal Nitrate test

The test was developed in 1933. During test time suspect hand was covered with wax. After the wax is completely settled down, take it out and take it for a chemical test. Then blue color will form indicating the presence of nitrites.

4. ADVANCED TECHNIQUE FOR THE IDENTIFICATION OF GUNSHOT RESIDUES

4.1 IDENTIFICATION OF INORGANIC GUNSHOT RESIDUES (IGSR)

4.1.1 Neutron Activation Analysis (NAA)

Neutron Activation Analysis is a nuclear process that can be used to detect gunshot residues by identifying the presence of barium and antimony on the hand of the shooter. The process determines the concentrations of elements in residues by bombarding the sample with neutrons to create radioactive isotopes. To examine gunshot residues on the hand of the shooter which has been collected by using the method of swabbing. It should be useful to attach small amounts of residues present on the suspect's hand. A cotton swab is moistened with 5% Nitric acid and rubbed against the suspect's hand. After that take this swab for testing the presence of barium and antimony in neutron activation analysis. It is the most effective technique. However, this method is not used routinely because of its complex nature, and experiences difficulties in collecting the voluminous background data required for the calculations. Neutrons are bombarded with the sample and convert it into a small compound which is highly unstable. These compounds pass through the intermediate state of being radioactive. This radioactive nucleus radiated delayed Gamma rays. The curve between the energy of gamma rays and radioactivity is marked and gives qualitative and quantitative data. [5] [6]

4.1.2 Atomic Absorption Spectroscopy (AAS)

Atomic Absorption Spectroscopy is an analytical method used to detect gunshot residues by measuring the amount of trace elements including lead, barium, and antimony. It cannot determine whether the person has discharged a firearm or not but it gives positive results in the detection of gunshot residues. The technique is very expensive and destructive. It is a very sensitive quantitative analysis method. The mechanism of this method is based on a very simple principle. The technique tests the concentration of gas-phase atoms in the sample. Those concentration were determined by the amount of light absorbed by free ions in the given sample. Then the sample will expose light at a specific wavelength to determine the presence of elements in the sample and also find its concentration [9]

4.1.3 Scanning Electron Microscope – Energy Dispersive Spectroscopy (SEM – EDX)

The SEM is equipped with an X-ray detector which can analyze X-ray spectra in a sample of a few cubic microns. This microscope utilizes a bond of electrons rather than promoting light to observe the

objects. Now the scanning electron microscope has been combined with energy-dispersive x-ray spectroscopy which is the most used technique for detection and chemical characterization of inorganic gunshot residue particles. It is a non-destructive technique. It can analyze particles left on the hand or any other surface after the person discharges the firearm, the identification includes micrometer-sized residue from the bullet, coating or jackets over the bullet, and composition of primer. Scanning Electron Microscopy can probe the microscale structures of particles from gunshot residues with high resolution to reveal morphological details that indicate fast-cooled droplets of molten materials that are the characteristics of gunshot residues. The combination of Energy Dispersive Spectroscopy was established to expose the compositional examination on individual particle level which allowed for not more than one interpretation detection of gunshot residue particles from given trace samples. The tape lifting method is most commonly used for collecting samples for SEM analysis. In this carbon adhesive tape was used to collect gunshot residue samples, and after collection take it for analysis in SEM. [10][14]

4.1.4 Inductively Coupled Plasma – Mass Spectrometry (ICP- MS)

This was the new logical system to determine the presence of lead, antimony, and barium from projectile residue patches from manual fusions, pellet and cartridge cases, etc. It can help to describe small projectile patches and dissect thousands of patches per nanosecond. It can also be used to prognosticate the number of shots fired from different arms and to estimate the origin of GSR by determining other rudiments like copper, nickel, and silver.[13]

4.1.5 Atomic Force Microscopy (AFM)

Atomic Force Microscopy is a nanoscale tool that can be used to study the morphological parcels of projectile residue patches. It has been used in ballistic division to study the 3-D geomorphology of fired cartridge cases and pellets and projectile residue patches on the shooter's hand. The GSR patches are collected using double-sided tape recording and taken for analysis in the microscope.

4.2 IDENTIFICATION OF ORGANIC GUNSHOT RESIDUES (OGSR)

4.2.1 Raman Spectroscopy

It's a non -non-destructive technique that can be used to dissect organic gunshot residues. Organic projectile remainders are made up of combustion products from arm discharge similar to nitrocellulose, nitroglycerine, nitroguanidine, stabilizers, plasticizers, and inert accouterments. Raman spectroscopy has only been used to identify a variety of different essence ions contained in GSR patches similar as oxide

fusions, sulfate, carbonate, barium chloride, lead oxide, lead sulfate, and iron oxides. Movable Raman microscopy is used to describe six organic projectile live analytes. Those analytes include diphenylamine, ethyl centrality, 2,4- dinitro toluene, 2- nitro diphenylamine, 4- nitro diphenylamine, and N- nitroso diphenylamine [17]

4.2.2 Attenuated Total Reflectance – Fourier transfigure Infrared Spectroscopy (ATR – FTIR)

The discovery of gunshot residues on the skin is the most important thing in forensic wisdom, so for that, downgraded Total Reflectance – Fourier transfigure Infrared Spectroscopy is used in arm examinations. It's a non-destructive fashion for the discovery of organic projectile remains. The remainder were uprooted from the shooter's hand by using the tape-recording lifting system. Before analysis add Na0 Ca bleach result to the all-projectile remainder samples on the tape recording for destroying the skin. The spectroscopic imaging fleetly and automatically scrutinized large areas of the tape recording- collected substrates and detected varying bitsy and macroscopic morphological features and chemical compositions from collected samples. All those characteristics are unique structures. The spectroscopic images targeted the discovery of those unique chemical labels over the area. [16]

4.2.3 Gas Chromatography-Mass Spectrometry (GC- MS)

It's a system used to describe organic projectile remainders and identify composites related to them. It can be employed to separate and describe the organic complements in handgun security fuel fusions and to dissect the substantiation related to smokeless grease paint composition. The GC- MS combination came in 1959. The fashion was employed to distance the admixture depending on the allocation between the stationary and mobile phases. GC- MS was set up to be used for the discovery of colorful explosive composites used in shots or security. [2] [4]

4.2.4 High-Performance Liquid Chromatography (HPLC)

High- Performance Liquid Chromatography fashion procedure for discovery and separation of organic projectile remainders. Prize the remainders and also dissect the methanol result with HPLC – MS. It's a fast and dependable system for relating diphenylamine. Diphenylamine is a successful target due to its oneness as a stabilizer in smokeless gunpowder. The system identifies organic projectile remainders including ethyl centralite, dimethyl phthalate, diphenylamine, 2- dinitro phenylamine, and 4- dinitro phenylamine.

5. OTHER TECHNIQUE FOR THE IDENTIFICATION OF GUNSHOT RESIDUES

5.1 Laser- Induced Breakdown Spectroscopy (LIBS)

Laser Induced Breakdown Spectroscopy supplies multi-elemental examination of the sample and needs veritably small traces of material and no sample medication. The system is used for the observation of GSR patterns. After collectively characterizing the GSR patches, measure the distribution of lead, barium, and antimony over the face of clothes, hands, hair, shot from different distances, etc. The GSR patches are collected by hand by dabbing with the help of tenacious videotapes. The GSRs were restrained from the fired charges by swabbing with cotton hearties. The tar is located in the LIBS sample chamber and dissolved in multiple shots at different tar spots. The cooperated system of Gas Chromatography coupled with Mass Spectroscopy and LIBS is recently suggested for the performance of both organic and inorganic GSR composites. This fashion helps to identify the isolation between shooter and non-shooter. [12]

5.2 Photoluminescence Technique

The photoluminescent projectile residue is examined for readily relates to space and position- resolved discovery of projectile remainders with high resolution. Lead dust in projectile residue reacts into a lead halide perovskite semiconductor that emits bright green light under ultraviolet irradiation. The veritably important response of photoluminescent lead allows strong identification of trace quantities of GSR. Photoluminescent lead determination yields reproducible projectile residue patterns for shooting distance reconstruction sequences. [7]

5.3 Ion Mobility Spectrometry

It's one of the most sensitive and robust ways for explosive discovery. In the early 2000s ion mobility spectrometry was used for the discovery of organic projectile remainders. It's used for the determination of smokeless maquillages exercising a collection sludge in combination with thermal desorption for the sample instigation. Nitroglycerin, nitrocellulose, and nitrate were also detected. The combination of solid-phase microextraction coupled with ion mobility spectrometry is used for the identification of unpredictable and semi-volatile complements of smokeless maquillages. This system was used for the analysis of organic projectile remainders which were collected with the help of cotton hearties from the shooter's hand. [5]

6. CASE STUDY

6.1 Analysis of Gunshot Residue Using Raman Spectroscopy

INTRODUCTION

The nitrocellulose and stabilizers were used as reference products for the visual comparison between the obtained Raman spectra. The unburned gunpowder samples were used for visualization in SEM and nitrocellulose was synthesized in the laboratory. In this study, a series of 36 test shots have been carried out on a test firing field using an automatic rifle Kalashnikova and semi-automatic handguns using two caliber types of national ammunition 7.62×39 mm and 9×18 mm respectively. All the cartridges were collected from the same batch of ammunition. Before firing, the shooter cleans the firearm to avoid cross-contamination. All shots were made at a muzzle target of 0° .

SPECTROSCOPIC ANALYSIS

The shooting distance was taken to maximize the number of organic gunshot residue particles obtained. Tissue strips of $8 \text{cm} \times 4 \text{cm}$ were cut from the surface of clothes after firing to facilitate the sample handling. A thermos scientific DXR Raaman microscope controlled by thermo scientific omnic for dispersive Raman software 9.6.249 was used for the analysis of organic gunshot residue particles. The microscope was set up to $50 \times$ magnification. The spectrometer was estimated before the Raman spectra were restrained using silicon. Each spectrum was an average of 10 scans for 10s and recorded in the range of 400-3500 cm⁻¹ for the visual comparison of spectra.

RESULTS

In this study, the most suitable measurement parameters were taken after performing so many tests. Mainly focus on the spectrum quality the time taken for sample analysis and the chances of organic gunshot residue samples decomposition. The most suitable laser wavelength allows reducing the light to be absorbed by the sample and allows the increase of the Raman signal intensity by a resonance effect. Due to the dark color of the samples, the 785nm laser source did not give satisfactory results for gunshot residue produced by 7.62 ×39mm, while 488 and 532nm laser wavelengths provide accurate results for both types of organic gunshot residue particles. A comparison between the Raman spectra of nitrocellulose and collected gunshot residue samples was obtained for two types of ammunition.



CONCLUSION

The obtained results based on the use of Raman spectroscopy analyses reveal the methodology as a potentially powerful tool for the detection and distinguishing between organic gunshot residues from such caliber containing different types of stabilizers. This permits to connection of the particles collected from the crime scene to a particular type of ammunition and firearm.[19]

6.2 Analysis of gunshot residues by SEM/EDX and NAA

INTRODUCTION

To collect GSR firing tests were performed using firearms such as Kalashnikova caliber 7.62×39 mm, shotgun Saint–Etienne 16, and semi-automatic handgun P. Beretta 7.65mm. The shots were taken on a special device called a water channel. The samples were taken by revealing places and according to the used weapons on the skin, both sides of hands, and tissue supports. The particles were collected with the help of white circular carbon adhesive tapes mounted on aluminum stubs.

NEUTRON ACTIVATION ANALYSIS

For the detection of gunshot residues, qualitative analysis was conducted. This was performed to determine the trace elements from the residues which contain four Levis supports of graphite, these represent the ideal matrix for the technique because graphite is transparent thermal neutrons. The four samples analyzed are called Geco-38sp/IRH, Geco-38sp/ERH, SERIANA -7.65mm/IRH, and SERIANA – 7.65mm/ERH. Each specimen was swaddled in aluminum of high purity. An empty envelope and two blank LSGs were also analyzed.

SCANNING ELECTRON MICROSCOPE ANALYSIS

The SEM microscope was coupled with an X-ray microanalyzer and equipped with a secondary electron detector. Automated classification and assistance with gunshot residue particles were carried out by the GSR software. The latter has a highly promising automatic calibration process that permits full and dynamic adjustment of the microscope column, the motorized stage, the backscattered analysis, and the x-ray analysis system. The specimens are arranged on the platinum in a regular or irregular pattern to more than 16 samples.

RESULTS

NEUTRON ACTIVATION ANALYSIS

The total calculations exhibit that only the components can be distinct from contaminants found on blank support and in envelopes. Due to its upgraded sensitivity, the technique analyzed only barium, antimony, zinc, bromine, and iron in most samples, all these purities are present in specific amounts in the graphite support. Because it does not interact with the neutron. Lead is not detectable by the technique because its reaction with neutron produces a radionuclide with a very short half-life.

SCANNING ELECTRON MICROSCOPE ANALYSIS

The morphological results showed that metal particles such as lead, antimony, and barium are from gunshot residue. The registration of a high concentration of gunshot residue particles on different supports especially on matrix ERH (class1) indicating the presence of PbSbBa and BaSb means the shooting was carried out from the right hand in which the outer surface is close to the ejection port and on matrix ERH (class 3) explaining that gun barrel with the left hand in which outer face is close to the muzzle end. The absence of GSR particles explains the fact that there is no ejection port which implies no exhaust smoke near the hand that operates the ballistic shot.

CONCLUSION

Neutron Activation Analysis with high sensitivity findings inorganic gunshot residues on the graphite sampling support. But it can't detect lead and also this technique is unable to detect whether these elements are from GSR or another source of pollution. The result of analysis and expertise gunshot residues by environmental scanning electron microscopy technique. The coordination of mineral particles acquired in the components of gunshot residues gives an idea about the sources of mineral constituents by visual identification of the origin. [18]

7. CONCLUSIONS

The conclusion is that chromatographic and spectroscopic analysis can help the investigators determine whether the suspected shooter may have fired a firearm or not and estimate the range of fire from muzzle to target. The forensic significance is that those techniques can help to detect the gunshot residue particles from the hand of the individual, shoot distances, and identify the residues on bullet holes. All those methods are non-destructive and some of them are cost-friendly, have high accuracy and it allows for re-examination. Still, there is no scientific method currently available to determine that the particular gun has been fired recently.

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