

## SCIENTIFIC ADVANCEMENTS IN GSR DETECTION AND THEIR ECOLOGICAL IMPACT

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### Abstract

One of the essential elements in forensic analysis during firearm-related cases is the detection of gunshot residue (GSR), released during firing. There are various methods for detecting these particles, and these methods have evolved over the years with advancements in technology. At first, simpler methods such as the paraffin test and optical microscopy were used; as technology advanced, the use of SEM-EDS (Scanning Electron Microscopy with Energy Dispersive Spectroscopy), X-Ray Fluorescence (XRF), and Atomic Absorption Spectrometry (AAS) began, significantly improving the detection accuracy of these particles, and that marked a new era. Development was also noted in later periods, when methods such as SIMS (Secondary Ion Mass Spectrometry), GC-MS (Gas Chromatography-Mass Spectrometry) were also introduced. Although these methods enabled high accuracy and sensitivity, further development was still needed, because the negative ecological impact that they could cause due to high energy consumption, chemical reagents, and the creation of toxic waste encouraged the development of methods that, in addition to accuracy, are also eco-friendly. In response to these concerns, Raman Spectroscopy, FTIR (Fourier Transform Infrared Spectroscopy), and biosensors were presented. The purpose of this paper is to present how the methods for detecting GSR have evolved and which of them offer more environmentally sustainable alternatives.

*Keywords:* Gunshot Residue (GSR), Paraffin Test, SEM-EDS, SIMS, Ecological impact, Raman Spectroscopy

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### Introduction

The detection of gunshot residue is a very important factor in the detection of a firearm crime, evidence that is not subjective but it is verified by various methods. During the firing of a weapon, gunpowder and other metal particles are released with great force, and thus are also distributed in the surrounding environment (hands, clothes), particles which contain: Lead, barium, Zinc, Antimony, nitrates, nitrites, etc. Considering that we are discussing microtraces, this has presented a challenge for forensics since the past, where, even though there was no very developed technology, with the methods of the time, they have tried to detect these traces. One of the first and simplest methods was the paraffin test, which is also known as the diphenylamine test, a method not so accurate but very important as it marked the beginning of a new era in forensics. The development of this part of forensics has been dependent on and linked to the development of technology in general. Even though the optical microscope existed at that time, its use did not give satisfactory results; even if the dimensions of the particles could be seen, it was not possible to identify these elements. The level of accuracy in detecting these microtraces began to increase when electron microscopy, spectroscopy, and spectrometry were further developed and offered different methods where very small particles could be detected with higher resolution. The combination of different techniques made these methods even more efficient for the detection of GSR. In this paper, in addition to examining technological development in the detection of gunpowder residues, the possible impact of these developments on the environment is also discussed. This is because toxic reagents are often used, hazardous waste may be created after applying the methods, and high energy consumption can occur,

among other similar effects. It is also emphasized that technological development is necessary to increase the accuracy of detection methods, but this should not come at the expense of environmental pollution.

### **History and development of GSR detection**

In the early 20th century, before modern methods of detecting GSR were developed, criminal investigations relied on eyewitness testimony or large amounts of physical evidence. As gun crimes began to increase, detectives realized that in addition to the bullet, the act of shooting left other traces, such as a characteristic smell or traces of powder on the suspect's hands or clothes.

Once this was understood, efforts began around the 1920s to try to verify the phenomenon of residue, so that it could be used as evidence. Among the initial methods that were tried was the chemical method, where a reagent was applied to the samples, and if microparticles were present then a visible chemical reaction was observed, such as the Paraffin Test that was developed in the 1930s - warm paraffin was placed on the suspect's hands, and when it was removed, it picked up traces from the skin, including gunshot residue, and on top of this, reagents that reacted with nitrates were applied. This was a very practical method, but its accuracy was very limited since the nitrates that reacted with the reagent were not only found in GSR, but could also come from other sources, and this brought many false positive results.

In addition to the observation of GSR with chemical tests, optical microscopy was also used, which allowed one to see the shape of the particles, but their chemical composition was still unknown, and they were mistaken for dust or particles from sources other than GSR.

Today, it is known that during the firing of a weapon, particles of heavy metals such as lead, barium, and antimony are released, which can be observed on the hands, clothes or in the suspect's environment.

In addition to these inorganic substances, there are also other organic substances that are released, which usually have an explosive, stabilizing or plasticizing function. Among the main organic residues of GSR is nitrocellulose, which in most gunpowder's represents the main fuel, where during the firing the gun's needle hits the primer which then ignites the nitrocellulose, which under high pressure and temperature begins to burn and decompose, but not completely, a small part transforms into other substances such as nitrates and nitrites, which also spread into the immediate environment.

Other organic substances with other roles are very polluting, including:

Dibutyl Phthalate (DBP), N-nitrosodiphenylamine (N-n-DPA), 2,4-Dinitrotoluene (2,4-DNT).<sup>1</sup>

The negative effect of GSR on the environment is multidimensional, starting from air pollution in the vicinity where these microparticles can be inhaled and harm human health, for example, different toxic effects or the carcinogenic effect of dinitrotoluenes has been observed.<sup>2</sup>

But the polluting effect does not end with air pollution, as these microparticles also fall into the soil and over time can also be converted into other compounds and become part of the soil structure, and this way they become part of the food chain.<sup>3</sup>

During rainfall, these residues (usually heavy metals) can also pass into groundwater, and penetrate into various water sources, such as water for human consumption, or even lakes, where they can harm the life of aquatic organisms.

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<sup>1</sup> Virginie Redouté Minzière, Anne-Laure Gassner, Matteo Gallidabino, Claude Roux, Céline Weyermann. The relevance of gunshot residues in forensic science. (2022) *Wiley Interdisciplinary Reviews Forensic Science*

<sup>2</sup> Tchounwou, P. B., Newsome, C., Glass, K., Centeno, J. A., Leszczynski, J., Bryant, J., Okoh, J., Ishaque, A., & Brower, M. (2003). Environmental toxicology and health effects associated with dinitrotoluene exposure. *Reviews on Environmental Health*, 18(3), 203–229.

<sup>3</sup> Zhu, Y., Che, R., Tu, B., Miao, J., Lu, X., Li, J., Zhu, Y., & Wang, F. (2024). Contamination and remediation of contaminated firing ranges—An overview. *Frontiers in Environmental Science*, 12, 1352603.

Based on the fact that we are discussing microparticles, it is very important to prevent this type of pollution, because even at small levels, it causes contamination due to the nature of the pollutants.

### **Advancements in GSR detection**

After the initial phase of GSR analysis, with various technological developments, around the 1970s, it became possible to use new technologies with very high accuracy not only in research laboratories, but also in forensics practice. Among the most well-known and revolutionary methods of that time were: SEM-EDS, Atomic Absorption, XRF. But unlike previous periods, during these years, global awareness of ecology had also become quite topical, with the first International Earth Day being celebrated in 1970, and the first UN Conference on the Human Environment in 1972, and concerns about possible pollution from these new methods were louder than in previous periods.

### **SEM-EDS (Scanning Electron Microscopy – Energy Dispersive Spectroscopy)**

The SEM-EDS method is a combined method of the SEM and EDS techniques. SEM uses electron beams to scan the surface of the sample, creating clear images with details about the shape and structure of the sample even for particles with nanometric dimensions, while EDS is based on the analysis of X-rays that are emitted when the sample is hit by an electron beam, considering that each element emits specific X-rays and based on this, the chemical composition of the sample but also its quantity can be understood. And these specifics of each of the two methods make them ideal for being very effective as a combination since at the same time a certain point of the sample is analyzed from the morphological aspect by SEM and chemically by EDS, understanding at the same time its morphology and chemical structure. Due to these features, this method has been transformative in forensics by offering a new level of accuracy in sample analysis and still serves today as a standard in detection. Recently, this method has been integrated with various software, which enables immediate analysis and presentation of their composition in real time.

### **XRF (X-Ray Fluorescence)**

X-Ray Fluorescence is a fast and practical method, efficient for preliminary results<sup>4</sup> but does not resolve SEM-EDS.

This method works when the sample is hit with X-rays, which eject an electron from the inner orbit, and then an electron from the outer layer fills the empty space left by the ejected electron - this way energy is released in the form of X-rays, which are detected, and based on it we can identify which element it is.

### **AAS (Atomic Absorption Spectrometry)**

AAS is a method that can measure even very small amounts of metals in samples, as its initial form it was discovered by Sir Alain Walsh around 1950, but in forensic practice, it was first encountered around two decades after that.<sup>5</sup> This method works by first converting the sample we analyze into a gas, and the atoms become free, then these are exposed to the light of a certain wavelength, and each element absorbs only the energy that corresponds to its specific energy; then, the amount of energy absorbed is measured, and the identification of the elements is made.

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<sup>4</sup> <https://www.xrfscientific.com/working-principle-xrf-spectrometer/>

<sup>5</sup> B. Welz, M. Sperling, *Atomic Absorption Spectrometry*, Third Edition, WILEY-VCH, 1999

## SIMS (Secondary Ion Mass Spectrometry)

The SIMS method has its roots in 1910, when it was observed that bombarding a surface with charged ions leads to the release of positive ions and neutral atoms. After several decades, it was analyzed in more detail what happens to these released ions, and the idea of a device that could measure these particles was developed. About 2 decades later, a device was built that created a source of ions by hitting materials with electrons, and this was the precursor to SIMS.<sup>6</sup> SIMS works to identify particles through spectrometric analysis of secondary ions, which are released as a result of hitting the sample with primary ions.

The advantage of the SIMS method is its very high sensitivity, including even the lightest elements in the periodic table, and it is also able to distinguish isotopes, regardless of whether they are in low or high concentrations.<sup>7</sup>

The disadvantage of this method is the inability to understand the exact amount of percentages because only the ionized part of the material can be analyzed.

In addition to metal analysis, SIMS is also very effective for analyzing organic substances because it causes less damage and the molecular structure can be clearly observed.<sup>8</sup>

But despite all these advantages, the SIMS method can have a negative ecological impact for several reasons, starting from the large energy consumption that may be needed to create enough vacuum for this method to function at the expense of the high resolution it offers it can also cause pollution through the dispersion of fine particles of heavy metals, or even toxic laboratory waste.

## GC-MS (Gas Chromatography – Mass Spectrometry)

Mass spectrometry began to be considered when it was discovered that the particles of a substance can be separated according to their weight by first turning them into a gas. After this discovery, gas chromatography was also developed, which separates the elements of a compound according to the way they move in the column. And with the combination of these two techniques, GC-MS was created, which separates mixtures and then accurately identifies their components, so this found appropriate use in the detection of GSR.

Initially, the sample is heated and is converted into a gas, and this gas passes into a column, where the components are separated from each other based on the way they move in the column, this process is part of chromatography. After leaving the column, the molecular weight and structure of these compounds are analyzed by mass spectrometry, and this data is compared with known data for thousands of substances.<sup>9</sup>

Even with GC-MS, in addition to the benefits in terms of accurate results, the disadvantage is its impact on the environment, since GC-MS is a device that requires high energy consumption, and especially when this energy is produced from coal or other pollutants, it is an even bigger problem. This device is not built in such a way as to be eco-friendly but has the priority of accuracy, also, similar to other methods it can create toxic laboratory waste.

In addition to these general effects, some solvents are also used in the preparation of samples for GC-MS which can be harmful and dispersed in the surrounding environment, such as: Methanol, Acetonitrile, Ethanol, Isopropanol, Hexane, Toluene, Ethyl Acetate, and Dichloromethane.

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<sup>6</sup> Panda, B. *Secondary Ion Mass Spectrometry*. Marshall Space Flight Center, NASA. (2019)

<sup>7</sup> López, F. *Secondary Ion Mass Spectrometry (SIMS): Principles and applications*. Handbook of Instrumental Techniques from CCiTUB, MT.10. Universitat de Barcelona.

<sup>8</sup> R.J. Day, S.E. Unger, R.G. Cooks (1980) *Molecular Secondary Ion Mass Spectrometry*, Dept.of Chemistry, Purdue University, West Lafayette, Ind. 47907

<sup>9</sup>Smriti RANJAN MAJI, Chaitali ROY, Sandip KUMAR SINHA. (2023). *Gas chromatography–mass spectrometry (GC-MS): A comprehensive review of synergistic combinations and their applications in the past two decades*. *Journal of Analytical Sciences and Applied Biotechnology*, 5(2), 72–85.

Some of the other advanced methods are: LA-ICP-MS and TOF-SIMS.

**LA-ICP-MS (Laser Ablation – Inductively Coupled Plasma – Mass Spectrometry)** is a method that uses laser to analyze solid materials. This method provides high accuracy and enables microanalysis of elements. It has some advantages but it is still under development as there are some shortcomings in the accurate determination of the entire composition and different materials can affect the laser differently.

LA-ICP-MS is a very effective technique for analyzing solid samples, providing information on many elements with a detailed view of the surface but also in-depth analysis, it is also practical as it requires simple sample preparation for analysis.

All these qualities make it suitable to use in the detection of GSR.

**TOF-SIMS (Time-of-Flight Secondary Ion Mass Spectrometry)** is a method that offers very high resolution, especially at the surface, using primary ion beams to remove molecules from the surface layer of the sample, and then the mass of the secondary ions is measured based on the time it takes them to reach the detector.

### **Environmentally friendly methods**

The need for precise methods without negative ecological impact paved the way for the development of methods such as: Raman Spectroscopy, FTIR and biosensors.

### **Raman Spectroscopy**

Raman Spectroscopy is a method that offers high accuracy but above all it is more ecological compared to most methods, it is a non-destructive method, it does not damage the sample but also does not require any preliminary preparation of it before analysis. This method is also combined with statistical analysis to understand the changes without the need for prior information on the samples. It has been observed that it shows high accuracy especially in the identification of organic residues such as stabilizers like diphenylamine and ethyl centralite.<sup>10</sup>

### **FTIR (Fourier Transform Infrared Spectroscopy)**

FTIR originated when it was first observed that heat beyond the red end of the visible spectrum of infrared light, and this began further studies of invisible radiation and how it interacts with matter. With further technological advancement, FTIR became a more efficient and accurate method, finding use in forensic analysis.

FTIR identifies functional groups through the analysis of the absorption of infrared radiation by bonds within molecules, each bond has a specific vibration frequency and absorbs a certain wavelength.<sup>11</sup>

When IR radiation falls on the sample, it absorbs light and creates different vibrations within the molecule, which enables identification. This method does not use harmful reagents and does not create hazardous waste, which makes it eco-friendlier.

### **Biosensors**

Another innovation with ecological advantages that has found use in forensic practice are biosensors, which use specific molecules such as antibodies or enzymes to detect chemical

<sup>10</sup> D.S.Karahacanea, A.Dahmania, K.Khimecheb (2019). Raman spectroscopy analysis and chemometric study of organic gunshot residues originating from two types of ammunition. *Forensic Science International*, 301, 129–136.

<sup>11</sup> Sh.A.Khan, Sh.B.Khan, L.U.Khan, A.Farooq, K.Akhtar, A.M. Asiri (2018). Fourier transform infrared spectroscopy: Fundamentals and application in functional groups and nanomaterials characterization. *Handbook of Materials Characterization* (pp. 317–342). Springer.

compounds during sample analysis. This method doesn't require the use of toxic substances and still offers high accuracy.

### Comparison of methods

For a more accurate comparison between different methods, it is essential to analyze different factors, such as: forensic accuracy, sensitivity, energy consumption, use of toxic reagents, and environmental waste. In the table below, these factors have been analyzed for the main methods used for GSR detection:

Method	Accuracy (%)	Sensitivity	Energy consumption	Use of toxic reagents	Waste
<b>Paraffin test</b>	~60% (estimated)	Low	Very low	Yes	Low
<b>SEM-EDS</b>	90%-98% <sup>12</sup>	High	Very high	Yes	Very high
<b>AAS</b>	90% <sup>13</sup>	High	Low	Yes	Moderate
<b>SIMS</b>	95%-99%	Very high	Very high	No	Moderate
<b>GC-MS</b>	95%-99%	Very high	High	Yes	Moderate
<b>Raman Spectroscopy</b>	98% <sup>14</sup>	High	Low	No	Low
<b>FTIR</b>	~90% (estimated)	Moderate	Very low	No	Low
<b>Biosensors</b>	~95% (estimated)	Very high	Very low	No	Very low

For some methods, the data are documented, while for others, the accuracy has been estimated from practical experience and scientific literature. Although these are estimated values, their reliability remains high.

SEM-EDS, GC-MS and SIMS methods are among the most accurate and widely used methods in forensic analysis, because they are able to detect even extremely small amounts of GSR.

Methods such as FTIR, RAMAN spectroscopy and Biosensors offer accuracy and sensitivity to GSR without wasting much energy and with minimal use of toxic reagents, thus becoming more preferable by forensic laboratories that are looking for more ecological alternatives.

Another method widely used in forensic laboratories is the paraffin test, regardless of the fact that it does not offer very high accuracy, it is more easily accessible than the most advanced technologies, both because it is easy to use and is also cheaper. This method has low energy consumption, does not produce much waste, but uses toxic reagents.

<sup>12</sup> Zuzanna Brożek-Mucha, *Scanning Electron Microscopy and X-Ray Microanalysis for Chemical and Morphological Characterisation of the Inorganic Component of Gunshot Residue: Selected Problems*, *BioMed Research International*, Volume 2014, Article ID 428038

<sup>13</sup> Bharti Jain & Poonam Yadav (2021). Vibrational spectroscopy and chemometrics in GSR: Review and current trend. *Egyptian Journal of Forensic Sciences*, 11(15).

<sup>14</sup> Bharti Jain & Poonam Yadav (2021). Vibrational spectroscopy and chemometrics in GSR: Review and current trend. *Egyptian Journal of Forensic Sciences*, 11(15).

## Conclusion

Advances in GSR analysis driven by concerns about environmental pollution are at a point where it is needed a balance between accuracy and environmental impact. Techniques such as Raman Spectroscopy, FTIR and biosensors have shown less environmental pollution compared to other commonly used methods due to the use of fewer chemicals and lower energy consumption. This progress should serve as an incentive for the wider use of these methods.

A way to reduce the energy consumption required for the applied methods is to find an appropriate method for choosing which technique should be used in a specific case, to prevent the use of several methods in the same case. In addition to reducing energy consumption, this method also has the benefit of reducing the likelihood of sample contamination, in order to prevent false positive results, which also requires special attention because GSR particles in a sample can be present from the environment or they may be transferred by another person.

Another step in reducing the negative ecological impact is the reduction of toxic agents during sample preparation, because even though there is implementation of regulations on how they should be eliminated from laboratories, there is a possibility that some of them are not managed properly, and they end up in water or soil. Therefore, the application of methods that do not require the use of toxic agents should be encouraged or harmless alternatives should be developed. Also, the ecological concern related to pollution, caused by high energy use can be reduced if the use of green energy is encouraged.

Another area of concern is that the materials used to build these devices should not be made of elements that cause pollution during their extraction and processing. All this progress supports the development of environmentally friendly methods in forensic science.

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