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Perovskite-based photoluminescent detection of lead particles in gunshot residue

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ABSTRACT

Gunshot residue (GSR) analysis is crucial for forensic investigations of shooting incidents, but current methods are time-consuming, laborious, and provide limited spatial resolution. We introduce photoluminescent lead analysis (PL-Pb) for instant high-resolution GSR detection. Lead particles in GSR react into lead halide perovskite semiconductors that emit bright green light under ultraviolet irradiation. PL-Pb enables trace detection of GSR, including ricochet markings, bullet wipes, and combustion plumes. Our method visualizes fine details like rifling patterns and facilitates and extends shooting distance reconstructions. We find that PL-Pb is also suitable for rapid presumptive testing of shooting suspect's hands, clothes, shoes, and other relevant objects. The instant results, sensitivity, and spatial resolution of perovskite-based detection of lead-containing micro-traces offer unprecedented opportunities for forensic investigations and environmental studies on lead particles.

1. Introduction

When a gun is fired, fine particles are released that typically contain lead [1,2]. These gunshot residue particles (GSR) not only cause health risks due to the toxicity of lead, but also offer opportunities for forensic investigations ranging from identifying shooters, to estimating shooting distances, and reconstructing shooting incidents at crime scenes [3–9]. Although lead-free ammunition is nowadays available, forensic experts almost exclusively encounter lead-containing ammunition due to low prices and wide availability [10–12]. Analysis of lead in GSR is therefore of great importance for forensics studies, but can ultimately also facilitate toxicological risk assessments.

Currently, lead in GSR is analyzed using either simple colorimetric reactions or sophisticated analytical techniques [1,9,11–20]. For investigating potential suspect involvement in shooting incidents, scanning electron microscopy (SEM) with energy dispersion spectroscopy (EDS) is often employed to detect highly characteristic GSR particles that contain lead, barium, and antimony which originate from the primer of ammunition rounds [6,7,11,19]. However, SEM-EDS for GSR analysis requires expensive, sensitive equipment, dust-free laboratory infrastructures, and specialized know-how. Additionally, processing EDS data can be laborious, thereby jeopardizing timely evidence production for legal processes. It also typically lacks spatial information (e.g. GSR patterns) due to the sampling process, thereby hindering activity-level analysis in shooting incidents.

For estimating shooting distances, identifying ricochet markings, bullet holes, and crime scene reconstructions, GSR is visualized using colorimetric reactions with rhodizonate [1,7,8,20,21]. Despite widespread use and extensive optimization, this test has several drawbacks. Rhodizonate may react with other components than lead and barium, resulting in false positives that complicate the forensic analysis [22,23]. Moreover, the color change can be difficult to interpret, especially under poor lighting conditions. Furthermore, direct analysis is often not possible on colored substrates and the applied chemistry is rather invasive. Therefore, a sampling step is often required. Hence, both for assessing shooting incident involvement of individuals and crime scene

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reconstruction analysis, there is a clear need to improve GSR characterization.

Building on a recent breakthrough [24,25], we here introduce photoluminescent lead (PL-Pb) detection as a fundamentally new GSR analysis method that relies on reacting lead particles into perovskite semiconductors that emit bright light. In recent years, perovskites gained tremendous attention for applications ranging from solar cells and detectors to catalysis and LED's [25–28]. Paradoxically, smaller perovskite particles can have brighter PL—likely due to favorable electron-hole pair recombination and improved reactivity—such that smaller lead particles may yield brighter PL. Leveraging this advantage, we detect lead particles in GSR and demonstrate the application potential and versatility by developing practical procedures for forensic scenarios such as shooting distance estimation, GSR transfer methods, rapid pre-screening of shooting incident suspects, and crime scene shooting incident reconstruction. These results highlight the potential for perovskite-based photoluminescent detection of lead containing, crime related micro-traces and create new opportunities for testing lead particles to assess environmental and toxicological risks.

2. Experimental section

To perform the experiments, we prepared the following reagents and equipment. The PL-Pb reagent, used for GSR visualization, was prepared using isopropanol and the perovskite precursor methyl ammonium bromide with additional additives. The transfer agent consisted of a 0.25 % w/v solution of benzoic acid in isopropanol (IPA). For the transfer substrate, we used a glass fiber cloth (Whatman™, CAT No. 1820-915). Compression was applied using a hydraulic press, set to $1.6 \cdot 10^6$ N/m^2.

For the firearms and ammunition, we utilized Glock 19 Gen5 and Walther P99Q NL pistols. Standard 9 mm full metal jacket bullets (S&B 9 mm LUGER V310492 FMJ) were used for all firing activities.

The experimental setup involved several steps. To create and deposit GSR, the firearms were fired at an unbleached cotton cloth placed at distances ranging from 0 cm to 200 cm (Fig. 1A). Visualization of the deposited GSR was achieved by illuminating the cloth under UV light (365 nm) and subsequently spraying it with a uniform thin film of the PL-Pb reagent using an atomizer spray (Fig. 1B). Since lead-containing GSR particles will be expelled upon the discharge of the weapon, all manner of surfaces can be tested, in this case hands, clothing, and the shooting targets themselves (Fig. 1C).

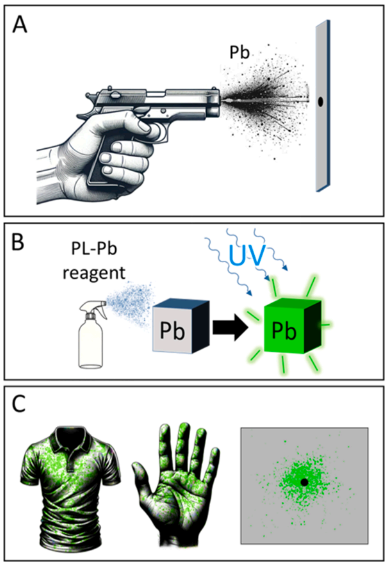

Fig. 1. Photoluminescent lead detection (PL-Pb) in gunshot residue (GSR). (**A**) Shooting firearms produces GSR that typically contains lead particulates. (**B**) Methyl ammonium bromide in the PL-Pb reagent reacts with lead in GSR to form perovskite, which emits bright green light upon illumination with UV light. (**C**) Typically, the combustion plume and bullet wipe contaminate objects such as clothing, hands, and targets with GSR, which is rapidly visualized with PL-Pb to assess suspect involvement and to assist crime scene reconstruction.

The transfer of GSR patterns was performed using the glass fiber cloth as the secondary substrate. The original GSR-deposited cloth was first sprayed with the transfer agent (0.25 % w/v benzoic acid in IPA) using an atomizer spray. The secondary substrate was then positioned on top of the treated cloth and compressed using the hydraulic press at $1.6 \cdot 10^6$ N/m^2. Once dried, the secondary substrate was sprayed with the PL-Pb reagent under UV irradiation to visualize the transferred patterns.

To estimate shooting distances, multiple shooting series were performed at distances ranging from 0 to 200 cm. GSR patterns from these series were transferred to glass fiber cloths and visualized with the PL-Pb reagent, allowing for comparative analysis.

Finally, presumptive testing for GSR detection was conducted on suspects and crime scenes. A modified non-toxic version of the PL-Pb reagent was directly applied to the hands of suspects. Additionally, GSR detection was carried out by swiping items such as hands, shoes, and clothing using a glass fiber swab.

3. Results and discussion

We assessed the compatibility of PL-Pb with GSR analysis (Fig. 2). A Glock 19 Gen5 and Walther P99Q NL pistol loaded with a standard 9 mm full metal jacket bullet (S&B 9 mm LUGER V310492 FMJ) was selected. Such a firearm and ammunition type frequently occur in forensic scenarios. GSR was created and deposited by shooting the Walther P99Q at a cotton cloth placed at 5 cm distance (Fig. 2B). To visualize GSR, we illuminate the cloth with UV light (365 nm) and apply a uniform, thin film of PL-Pb reagent (based on isopropanol and the perovskite precursor methyl ammonium bromide with further additives) using an atomizer spray (Fig. 2A). Immediately, the cloth reveals a well-defined bright green luminescent pattern, which is clearly visible to the naked eye (Fig. 2C). Also, in the absence of UV light, we observe a persistent yellowing of the white cotton that is consistent with the formation of lead perovskite and lead bromide, showing that PL-Pb can also function as a colorimetric test (Fig. 2D). Overall, even though the combustion products and presence of other metals may complicate perovskite formation and PL, visualization of GSR is successful and consistently results in bright PL.

Transfer of GSR patterns to a secondary substrate is desirable for many forensic applications to create minimally invasive tests. This prevents the direct application of invasive reagents to evidence materials and facilitates optimal testing conditions by using well-defined substrates. PL-Pb analysis is fundamentally different from currently used rhodizonate tests. We therefore develop a new GSR transfer method that is optimal for PL-Pb testing (Fig. 3). Specifically, the transfer agent and secondary substrate were tuned for the PL-Pb test to optimize the workflow, transfer fidelity, and PL sensitivity. For the secondary substrate, a glass fiber cloth (Whatman™, CAT No. 1820-915) of approximately 25 cm × 25 cm was selected that is non-fluorescent but strongly adsorbs lead [29], and is wettable by the transfer reagent, thus maximizing transfer while limiting undesired smearing to ensure high transfer integrity. Moreover, the surface of the cloth has limited structural features that can disturb the transfer. For the transfer agent, a solution of 0.25 % w/v benzoic acid in isopropanol (IPA) was selected. This transfer agent dries fast, causes minimal smearing, and stabilizes PL for optimal visualization of transferred GSR.

We note that traditional transfers for rhodizonate testing are labor-intensive due to the slow evaporation of water (ca.1-5 min) and long pressing time (ca. 2 min). In contrast, our transfer for PL-Pb is faster as it does not require a drying step and short pressing time (< 45 s). Comparison of the GSR on the original cloth and secondary transfer shows that the microscopic pattern is well-preserved (Fig. 3B–G). Around the

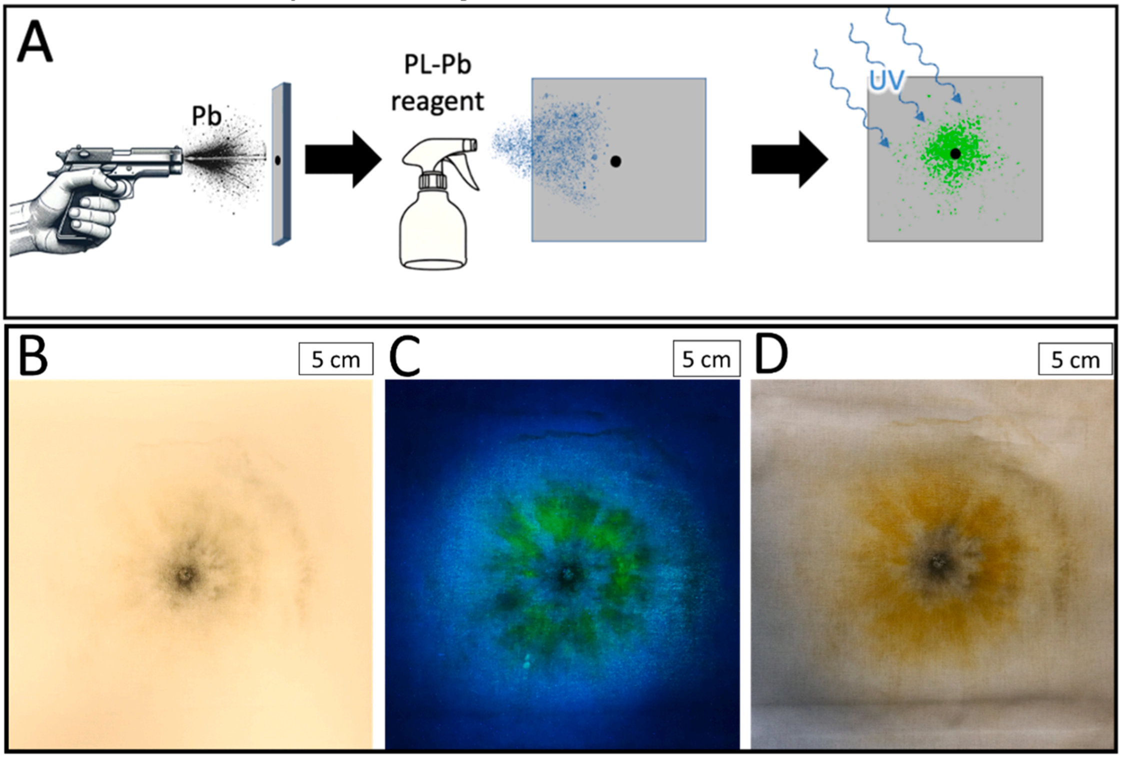

Fig. 2. Visualizing GSR with PL-Pb. (**A**) Procedure for visualizing lead in GSR using PL-Pb. (**B**) GSR is deposited by firing upon a cotton target at 5 cm distance. (**C**) Application of the PL-Pb reagent causes lead in GSR to emit green light under UV, while the cloth fluoresces blue. (**D**) Under visible light, yellow/orange colored lead halide perovskite and lead bromide indicate the presence of GSR.

bullet entry hole, we find a polygonal pattern, which matches the hexagonal rifling of the pistol. Even the micrometer fiber structure of the original substrate is resolved, which emphasizes the robustness and trace pattern integrity of the novel PL-Pb test.

We ascertain whether the sensitivity and integrity of PL-Pb analysis can be exploited for shooting distance estimations. Typically, shooting distance measurements are performed by shooting a firearm on substrates at different distances (Fig. 4A). Following this procedure, multiple shooting distance series were created ranging from 0 to 200 cm shooting distance (d) using a Glock 19 Gen5 pistol with 9 mm full metal jacket bullets (Fig. 4A, see SM for details). The resulting GSR patterns were transferred to glass fiber cloths following the optimized transfer method, and PL-Pb reagent application resulted in clearly discernable PL patterns (Fig. 4B). At $d = 0$ cm, we see tightly clustered GSR patterns [20]. At $d = 5$ cm, this cluster spreads out and we observe a polygonal pattern that is consistent with the barrel riffling. At $d = 10$ cm we observe circular rippling patterns that are consistent with pressure waves from the muzzle blast. At $d = 25$ cm, we record patterns corresponding to the combustion plume. Beyond $d = 50$ cm, these patterns are not observable, suggesting that the combustion plume is not reaching the substrate anymore (see Supporting information SI). Instead, a speckling pattern appears in addition to a clear bullet wipe at the entry hole. Surprisingly, even at $d = 200$ cm a speckling pattern and bullet wipe remain clearly visible. Compared to traditional shooting distance estimations with rhodizonate, we find that distance-dependent PL-Pb patterns are consistent with previous reports while extending the estimation range of the shooting distance. To test the reproducibility, the shooting series was repeated three times (see SI). We find similar pattern features at the same distances, hence showing that this distance measurement protocol provides reproducible results. Previous studies have shown significant variability in the quantity of GSR released from one shot to another. This can be affected by many variables, including weapon handling, environmental conditions, and even handedness of the shooter [30]. Additionally, personal communications with GSR experts from the *Netherlands Forensic Institute (NFI)* have indicated that the quantity of GSR particles expelled from a particular weapon can vary upwards of 500–600 % [31]. Despite this, distinct patterns consistently emerge depending on the distance between the firearm and the target. By observing these distance-dependent patterns during visualization, we demonstrate the consistency of this technique and its reliability for application in shooting investigations.

Next, we explore the use of PL-Pb for rapid presumptive testing of suspects and crime scenes (Fig. 5). The reagent is only mildly irritating to the skin, such that we can directly test hands of suspects [31]. Upon spraying the hand of a shooter, bright PL is observable on the entire hand (Fig. 5B), offering a clear and immediate method for presumptive GSR detection.

Detection of GSR after washing of hands is typically difficult as particles are removed [11,32], but can be relevant for forensic studies as suspects may try to tamper, conceal, or eliminate evidence of their involvement in a shooting incident. Even after extensive washing of the shooter's hands, we still detect PL, likely because of the regular shooting activities of the shooter and the sensitivity of the PL-Pb test (Fig. 5B, see SI for details). Bystanders positioned approximately 2 m from the shooter were also tested. Before shooting, their hands test negative. However, after the shooter fires ten times and the bystanders helped set up the target at different distances, the hands of bystanders emit a clearly visible PL signal (Fig. 5C), and even after casual washing and re-testing, a moderately heterogeneous PL signal persists (Fig. 5D). Extensive washing and scrubbing still results in a weak, heterogeneous PL signal focused on areas such as the fingernails and skin creases on the backside of fingers (Fig. 5E). Unlike traditional GSR methods, PL-Pb still detects Pb residues even after attempts to remove the GSR. Like the luminol test for blood, our method appears capable of detecting latent GSR traces.

We find that GSR is detectable on shooters and bystanders by swiping

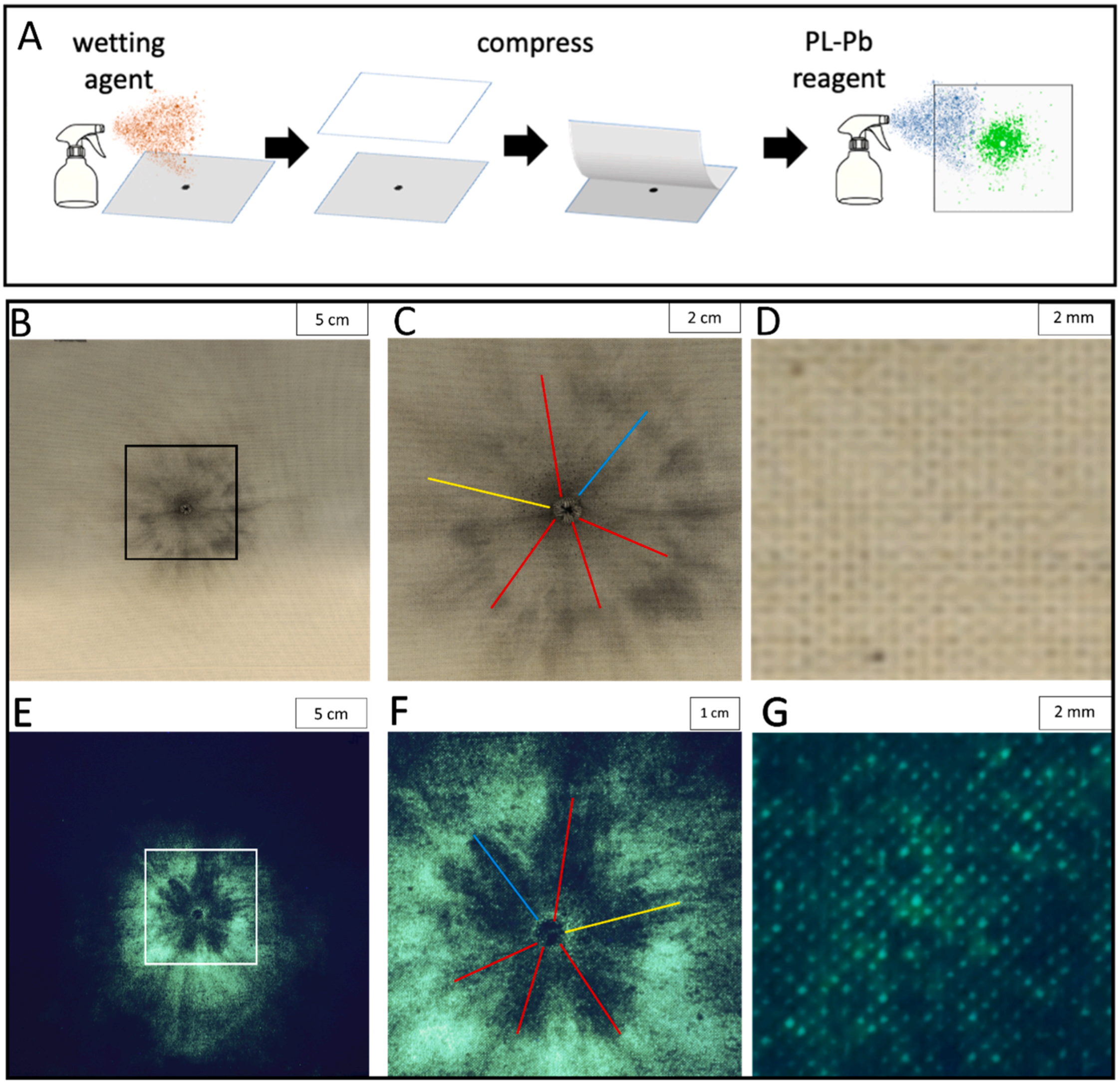

Fig. 3. GSR pattern transfer to a secondary substrate. (**A**) The original shooting cloth is sprayed with wetting agent (0.25 % w/v benzoic acid in isopropanol) and pressed onto a glass fiber cloth. (**B–D**) Original shooting cloth, showing increasing detail, and (**E–G**) corresponding transfer on secondary substrate after PL-Pb reaction. (**C**, **F**) Polygonal patterns from the barrel rifling are well-resolved (matching color markings highlight corresponding, mirrored, pattern features). (**D**, **G**) Submillimeter features of the shooting cloth are clearly visible on the secondary substrate, demonstrating the trace transfer pattern integrity with PL-Pb.

hands, shoes, clothing, or other items on a crime scene (e.g. steering wheels from cars, pockets in jackets etc.) with a glass fiber swab that is subsequently used to perform PL-Pb testing (Fig. 5F, G). Although spatial location of GSR may be lost by swiping, the advantage of this indirect testing method is that potential irritation or discoloration by the reagent can be avoided. Moreover, indirect testing limits potential interference from backgrounds (e.g. sweaty hands, fluorescent clothing), does not limit other forensic investigations, and can further lower the detection threshold through GSR accumulation.

While detection of Pb itself is not conclusive for GSR (particles containing lead may also originate from sources not related to a shooting incident), PL-Pb may serve as an indicative test for pre-screening of EDS stubs to expedite forensic work. To assess this potential, we stub the shooter's hand with carbon tape used for EDS analysis. We apply the PL-Pb reagent and directly detect PL from lead particles on the stub (Fig. 5H). Although compatibility with EDS analysis will require further research—in particular because dried reagent may complicate EDS particle counting software—these results suggest that PL-Pb testing can be valuable as pre-screening of shooting suspects. The main benefit of PL-Pb prescreening is that police officials immediately receive important information regarding a potential shooting incident. It can also boost efficiency in the forensic workflow by ranking samples for SEM-EDS analysis in the forensic laboratory. However, this does require more insight in the selectivity and sensitivity of PL-Pb vs SEM-EDS and tailor-made sampling strategies that ensure that the SEM-EDS analysis is not obstructed or limited in any way.

Here, we introduce a new GSR analysis technology based on the formation of lead halide perovskite semiconductors that emit bright light. Already, forensic photoluminescent detection techniques for samples such as blood and sperm are well-established, and forensic teams extensively use sophisticated UV-light-sources. We therefore anticipate that PL-Pb detection is highly compatible with current forensic workflows. In addition, our rapid transfer protocol combined with instant detection may speed up labor-intensive forensic practices and thus decrease this time-consuming bottleneck currently seen in GSR analysis. Further research on this workflow is currently in progress.

The instant, robust, and sensitive results of PL-Pb on the hands and clothing of shooters and bystanders open new opportunities for presumptive testing. Despite its non-toxic nature, the methylammonium bromide in the reagent is corrosive. We therefore believe that indirect testing using for instance swabs or cloth will be most attractive. First responders such as police officers can use the PL-Pb test to rapidly screen

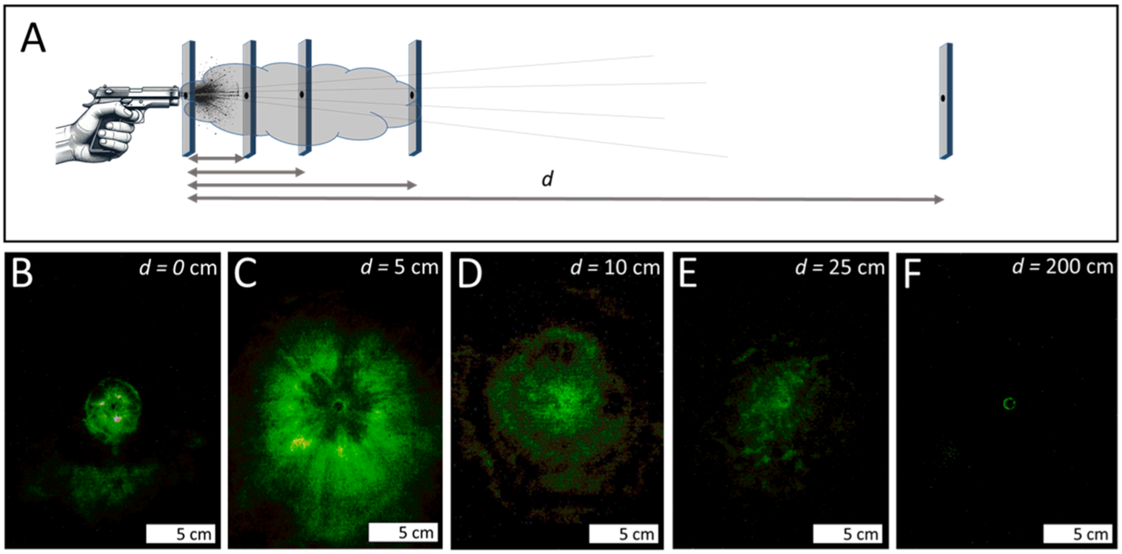

Fig. 4. Shooting distance analysis using PL-Pb. (**A**) Setup for creating shooting distance series at distance d. Transferred GSR shooting distance patterns, showing for (**B**) $d = 0$ cm tightly packed patterns, (**C**) $d = 5$ cm polygonal patterns corresponding to the barrel rifling, (**D**) $d = 10$ cm rippling patterns of concentric rings consistent with the infrasonic compression waves, (**E**) $d = 25$ cm patterns from the combustion plume, and (**F**) $d = 200$ cm speckling pattern encompassing the bullet wipe. See SI for full series and reproductions.

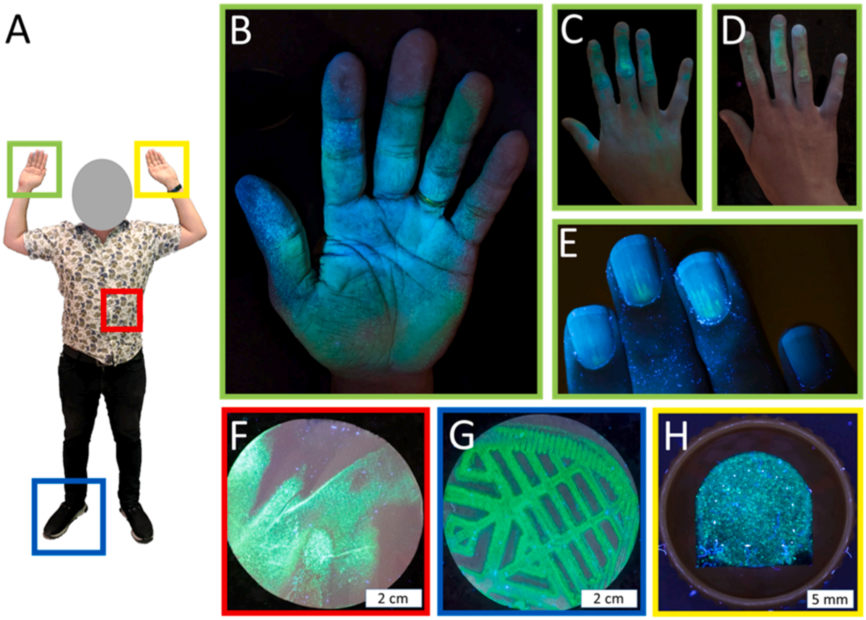

Fig. 5. Assessing suspect involvement in shooting incidents using PL-Pb. (**A**) Color marked areas for GSR testing. (**B**) Direct PL-Pb testing on shooter's hand showing bright PL. (**C**) PL-Pb testing on the hand of bystander after shooting showing moderate PL, and (**D**) weak PL after washing with water and soap. (**E**) Bystander's hand after thorough washing still showing clear PL-Pb on nails. (**F**) Indirect PL-Pb testing by wiping glass fiber cloth over clothing of a bystander shows bright PL. (**G**) Indirect testing of bystander's shoe sole. (**H**) SEM stub after stubbing shooter's hand showing PL-Pb.

potential suspects and witnesses or define crime scenes to secure evidence, thus providing an opportunity to address the overflow of samples sent for SEM-EDS testing. We are currently investigating how such prescreening can be performed.

The testing method can be tailored for specific scenarios. For example, background fluorescence coming from clothing may hinder signal interpretation. Currently, we overcome these complications by transferring GSR to non-fluorescent substrates. However, tuning of the reagent may enable red shifting of the excitation light source from UV towards the visible spectrum such that backgrounds do not show fluorescence while the perovskite still emits light. Furthermore, analysis of samples in a miniaturized dark-box in combination with image analysis software may be helpful for standardizing and optimizing automated PL-Pb characterization. Additionally, future studies will include comparison studies of current techniques, such as sodium rhodizonate for visualizing shooting patterns and preliminary testing at the crime scene, while also investigating this technology's cross-compatibility to other pieces of evidence, such as DNA traces. Furthermore, quantitative studies regarding previously mentioned correlations between particle size and testing sensitivity will be tested in the future.

Beyond forensic work, our PL-Pb detection method can immediately impact the identification of health hazards caused by lead in GSR [3,4]. Lead is a potent toxin, inflicting a wide range of large and irreversible health threats ranging from behavioral problems, aggression, and learning disabilities, to severe physical illnesses such as blindness, convulsions, cardiac diseases, and—ultimately—death [33–36]. Since lead does not degrade, it accumulates in areas such as shooting ranges. Hence, detection of lead in GSR is of importance for limiting health risks for regular shooters and bystanders such as military personnel, police officers, and operators at shooting ranges [4]. During this study, we found that PL-Pb effectively identifies lead in GSR at shooting ranges, suggesting that PL-Pb protocols could help mitigate lead poisoning risks in these environments.

More broadly, this study highlights the application potential of perovskite formation for lead detection. Lead in GSR is formed as micrometer fine dust particles. Ingestion of lead in any form or concentration is known to be dangerous [3,4,37]. Lead dust is a particular risk as it can be inhaled or ingested, but practical detection of lead dust is still difficult. While generally smaller particles become harder to detect, paradoxically, for our test, smaller particles may become easier to detect as they emit brighter PL. This increase in sensitivity for smaller lead particles suggests a unique strength of our perovskite-based lead detection, which may be further exploited for detecting lead dust from other sources as well. More generally, this study emphasizes the potential of perovskite formation for lead detection in scenarios that are of societal relevance. For instance, detection of low concentrations of lead in water, and biological samples such as blood is still challenging, but is essential to prevent and mitigate lead poisoning. We foresee that lead detection strategies based on perovskites formation can revolutionize this field, ultimately empower professionals and communities to ensure a safe environment for everyone.

Significance Statement

Shooting incidents carry significant societal impact, highlighting the importance of the accurate forensic investigation and reconstruction of these events. Identifying the shooter, determining the firearm type, linking ammunition parts to a firearm, and determining the location where a shot was fired are of crucial importance, but to date remain surprisingly difficult to establish. This research introduces a method to significantly advance these inquiries by converting lead-containing gunshot residue (GSR) into light-emitting perovskite semiconductors. The ease-of-use, sensitivity, and fidelity to resolve GSR patterns makes this perovskite-based photoluminescent detection directly suitable for integration in current forensic investigations. Furthermore, this study underscores the potential of perovskite-based detection of lead-containing micro-traces in forensic studies and for evaluating environmental risks associated with toxic lead.

CRediT authorship contribution statement

Adelberg Kendra: Writing – original draft, Methodology, Investigation. **Helmbrecht Lukas:** Writing – review & editing, Methodology, Investigation, Conceptualization. **van der Weijden Arno:** Writing – original draft, Methodology, Investigation, Conceptualization. **van Asten Arian C.:** Writing – review & editing, Supervision, Methodology, Conceptualization. **Blaauw Diede:** Investigation. **Noorduin Willem L.:** Writing – review & editing, Writing – original draft, Supervision, Methodology, Investigation, Funding acquisition, Conceptualization.

Declaration of Competing Interest

A patent has been filed related to the topic covered in this publication. LH and WLN are co-founder and co-owner of Lumetallix B.V., a company for lead detection. The remaining authors declare no competing interests.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.forsciint.2025.112415.

Data and materials availability

All data are available in the main text or the Supplementary materials.

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