Photoluminescent detection of lead dust in gunshot residue

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Abstract

Gunshot residue (GSR) analysis is essential for the forensic investigation of shooting incidents, but oftentimes still slow, cumbersome, and with limited spatial resolution. We here introduce a photoluminescent gunshot residue analysis (PL-Pb) for instant spatially resolved detection of GSR with high resolution. Lead dust in GSR reacts into a lead halide perovskite semiconductor that emits bright green light under ultraviolet irradiation. The sensitivity of PL-Pb enables straightforward detection of trace amounts of GSR from ricochet markings, bullet holes and combustion plumes. We show that GSR is transferable with high spatial resolution and preservation of fine details such as the polygonal patterns caused by the rifling of the pistol. Moreover, PL-Pb detection yields reproducible GSR patterns for shooting distance reconstruction series. We also show that the sensitivity and instant results make PL-Pb suitable for rapid presumptive testing of shooting suspects. Surprisingly, even after washing, we can still detect GSR on the hands of shooters, and we find GSR in a simple and straightforward manner on clothes, shoes, and other objects relevant to a shooting incident. Collectively, the instant results and sensitivity of PL-Pb opens unprecedented opportunities for (on-site) forensic investigations and highlights the potential of perovskite-based lead detection methods for lead containing, crime related micro-traces and lead dust in general.

Keywords: gunshot residue, perovskite semiconductors, photoluminescent lead test, forensic science, lead dust, lead pollution

Introduction

Shooting guns releases dust particles, which generally contain lead (Figure 1).[1,2] The lead dust in these gunshot residues (GSR) not only causes health risks due to the toxicity of lead, but also offers opportunities for forensic studies ranging from identifying shooters, to estimating shooting distances, and crime scene reconstructions.[3–9] Although lead-free ammunition is available, forensic scenarios almost exclusively encounter lead-containing ammunition due to low prices and wide availability.[10–13] Analysis of lead in GSR is therefore of great importance for forensics studies, but can ultimately also impact detection of lead dust for assessing toxicology risks.

Currently, depending on the forensic case context, lead in GSR is analyzed using either simple coloring reactions or sophisticated analytical techniques.[1,9,12–21] For investigating the potential involvement of suspects in a shooting incident, GSR is sampled by "stubbing" the hand of the suspect with a carbon sticker, which is then analyzed using scanning electron microscopy (SEM) equipped with energy dispersion spectroscopy (EDS).[6,7,12,19,22] SEM-EDS detection and elemental analysis of particles is considered the gold standard in the forensic GSR analysis. Especially particles containing the elements Pb, Ba, and Sb, originating from the primer in an ammunition round, are considered to be highly characteristic for residues released after firing a firearm. However, routinely applying SEM-EDS for GSR analysis requires expensive, sensitive equipment, a dedicated, dust-free laboratory infrastructure, and trained experts. Additionally, processing of EDS results can be laborious and can take days or much longer, in case of high caseloads. The resulting backlog can even jeopardize timely evidence production for legal processes.[23] Another limitation of GSR particle analysis with SEM-EDS and the associated sampling process is that no information is provided on the activities and the roles of those involved in a shooting incident. As a result, a forensic GSR expert typically cannot differentiate a shooter from an innocent bystander on the basis of SEM-EDS analysis alone. For activity-level analysis forensic GSR experts need to resort to more invasive colorimetric methods to visualize the GSR pattern (location, amount, shape).

For estimating shooting distances, identifying ricochet markings, bullet holes, and similar crime scene reconstructions, GSR is analyzed using colorimetric reactions with rhodizonate.[1,7,8,24,25] Rhodizonate changes color from yellow to brown upon complexation with lead and barium from the GSR. Despite widespread use and extensive optimization, this test has major drawbacks. Depending on the conditions, rhodizonate test can react not only with lead, but also with for instance barium, bismuth, cadmium, copper, mercury, and titanium, as well as to the keratinous structures of hair follicles and lingual papillae, which may result in false positives and complicated or inconclusive patterns.[26,27] Moreover, the color change can be difficult to interpret, especially under poor lighting conditions. Hence, both for assessing shooting incident involvement of individuals and crime scene reconstruction analysis there is still much room for improvement in current GSR characterization methods.

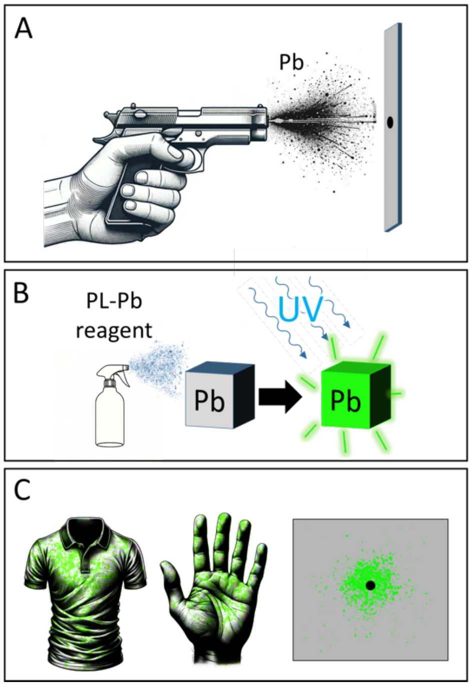

Figure 1: Concept of photoluminescent lead detection in gunshot residue (PL-Pb). (A) Shooting firearms produces GSR that typically contains lead particulates. (B) PL-Pb reagent reacts with lead in GSR to form perovskite, which emits bright green light upon illumination with UV light. (C) Typically, the combustion plume and bullet wipe contaminate objects such as clothing, hands, and targets with GSR, which is rapidly visualized with PL-Pb for suspect identification and crime scene reconstruction.

We propose a fundamentally different approach for detecting lead in GSR. The key idea is to turn lead in GSR into a lead halide perovskite semiconductor that emits bright green light under ultraviolet (UV) irradiation (Figure 1B). We recently exploited the versatile chemistry of lead and the bright photoluminescence (PL) of lead halide perovskites to develop a lead testing method based on perovskite formation.[28–31] In short, direct application of a reagent containing perovskite precursors (e.g. methylammonium halides) readily reacts with lead to form a lead halide perovskite semiconductor. Under UV-light, the perovskite shows strong photoluminescence in the visible range with a tunable wavelength, which makes lead detection straightforward and highly sensitive. We extensively benchmarked the sensitivity and selectivity and found that both false positives and false negatives were highly unlikely, while even 1 ng of lead can be detected readily by the naked eye. Moreover, we showed that this perovskite-based lead test is at least 1000 times more sensitive compared to state-of-the-art rhodizonate tests under laboratory conditions.[28] Unexpectedly, despite the notoriously problematic stability of perovskites, we detected lead in objects such as paints, waterpipes, dust, glass, and plastic, demonstrating that testing in real-world environments and conditions is feasible in a robust and reliable manner.

Motivated by these insights, the objective of this study is to determine how photoluminescent lead testing can be suitable for GSR analysis. Besides demonstrating a proof-of-principle, it would be helpful if such a new perovskite-based method is applicable in a range of relevant forensic scenarios where GSR analysis currently is challenging, such as fast and sensitive prescreening of GSR samples from crime scenes and potential suspects. Additionally, such a test could also be of value to select samples for further analysis with SEM-EDS in the forensic laboratory, leading to a better use of scarce expert and instrument capacity and an increased efficacy when considering the overall case load.

We here demonstrate the proof-of-principle of photoluminescent GSR (PL-Pb) detection for instant spatially resolved detection of GSR with high resolution and sensitivity (Figure 1). Paradoxically, smaller perovskite particles can have brighter PL—likely due to favorable electron-hole pair recombination—such that smaller lead dust may yield particles with brighter PL. This suggests that, in contrast to traditional techniques, finer lead dust may be easier to detect by perovskite-based analysis. Leveraging this advantage, we show the application potential by developing practical procedures for forensic scenarios such as shooting distance estimation, GSR transfer methods, rapid prescreening of shooting incident suspects, and crime scene reconstruction (Figure 1C). We also show that Pb-PL can be suitable as indicative test for further analysis with SEM-EDS in the forensic laboratory. These results highlight the potential for perovskite based photoluminescent lead detection methods for lead containing, crime related micro-traces and open new opportunities for testing lead dust to assess environmental risks of toxic lead.

Results and discussion

We assess the compatibility of the perovskite-based photoluminescent lead test with GSR analysis (Figure 2). To demonstrate the proof-of-principle, we discharge a pistol and react the released GSR with the PL-Pb reagent (see Supporting Information (SI) for details). The PL-Pb reagent is a proprietary mixture from Lumetallix based on the solvent isopropanol and the perovskite precursor methyl ammonium bromide. The PL-Pb reagent limits interference of other combustion products, while simultaneously producing enduring and clearly visible bright green PL for optimal forensic work.

We select 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). Such weapon and ammunition types commonly occur in forensic scenarios. To deposit GSR, we shoot the Walther P99Q at a bleached, fluorescent cotton cloth placed at 5 cm distance (Figure 2B). To visualize the GSR, we illuminate the cloth with UV light (365 nm) and subsequently mist it with a uniform thin film of PL-Pb reagent using an atomizer spray (Figure 2A). Immediately, the cloth reveals a well-defined bright green luminescent pattern, which is clearly visible to the naked eye (Figure 2C). Also, in the absence of UV light, we observe a persistent yellowing of the white cotton which is consistent with the formation of lead perovskite and lead bromide, showing that PL-Pb can also function as a colorimetric test (Figure 2D). Overall, even though the combustion products and presence of other metals may complicate the perovskite formation and PL, we find that visualization of GSR is successful with bright PL.

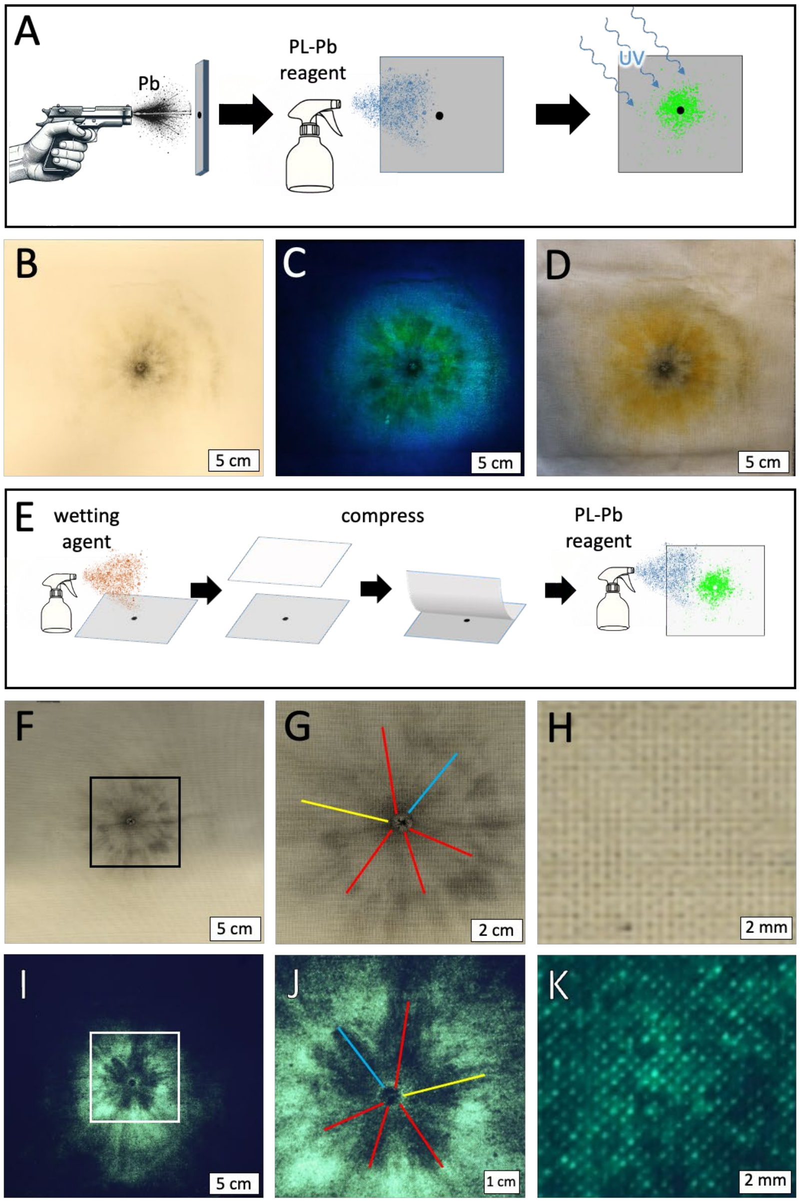

Figure 2: Photoluminescent lead detection in GSR. (A) Procedure for visualizing lead in GSR by applying the PL-Pb reagent under UV-light. (B) Using a Walther P99Q NL with standard 9 mm full metal jacket bullets, GSR is deposited by firing upon a cotton target placed at 5 cm distance. (C) Upon application of the PL-Pb reagent, lead in the GSR lights up green under UV light while the cloth lights up blue due to auto-fluorescence. (D) Under visible light, the GSR is visible due to formation of the yellow/orange colored lead halide perovskite and lead bromide. (E) To transfer the GSR pattern to a secondary substrate, the original shooting cloth is sprayed with the wetting agent (0.25% benzoic acid in isopropanol) to facilitate the transfer of lead, and pressed on a glass fiber cloth with a force of $1.6 \cdot 10^6$ N/m^2 for 30 seconds. (F, G, H) Original shooting cloth created by discharging a Glock 19 Gen5 at 5 cm distance, showing increasing levels of detail, and (I, J, K) corresponding transfer on secondary substrate after PL-Pb reaction. (G, J) Polygonal patterns from the barrel rifling are well resolved with PL-Pb (matching color markings highlight corresponding pattern features). (H, K) The submillimeter structure of the shooting cloth is clearly visible on the secondary substrate, demonstrating the transfer fidelity achievable with PL-Pb.

Transfer of GSR patterns to a secondary substrate is desirable for many forensic applications, in particular to create a minimally invasive test—as reagents are not directly applied to the evidence materials, and because testing conditions are more controllable with well-defined substrates. Because our PL-Pb analysis is fundamentally different from currently used rhodizonate tests, we here develop a new GSR transfer method that is optimal for PL-Pb testing. Specifically, we tune the transfer agent and secondary substrate for the PL-Pb test to optimize the workflow, transfer fidelity, and PL sensitivity. For the secondary substrate, we select glass fiber cloth without binder sourced from Whatman and Macherey-Nagel. This cloth is non-fluorescent, strongly adsorbs lead,[32] and is wettable by the transfer reagent, thus maximizing transfer while limiting undesired smearing for high transfer fidelity. Moreover, the surface of the cloth has limited structural features that can disturb the transfer. For the transfer agent, we select a 0.25% solution of benzoic acid in isopropanol (IPA). This transfer agent can partially dissolve the lead particles, dries fast, causes minimal smearing, and stabilizes PL for optimal visualization of the transferred GSR.

To perform the transfer and GSR detection, we shoot at a cotton cloth using a Glock19 Gen5 at 5 cm distance (Figure 2F). We transfer the deposited GSR pattern by spraying the original substrate with the benzoic acid solution using an atomizer spray, positioning the secondary substrate on top, and apply $1.6 \cdot 10^6$ N/m^2 compression using a hydraulic press (Figure 2E). Once detached, the secondary substrate is sprayed with the PL-Pb reagent using an atomizer spray under UV irradiation (Figure 2I). Comparison of the GSR on the original cloth and secondary transfer shows that the microscopic pattern is well-preserved (Figure 2F-K). Around the bullet entry hole, we find a polygonal pattern, which matches the hexagonal rifling of the pistol. Closer inspection of the secondary substrate reveals that we can even resolve the micrometer features of the fiber structure of the original substrate, which emphasizes the conservation of original GSR patterns with the PL-Pb test. We note that, traditional transfers for rhodizonate testing are labor-intensive due to the slow evaporation of water (ca.1-5 minutes) and long pressing time (ca. 2 minutes). In contrast, our transfer for PL-Pb is faster as it does not require a drying step and short pressing time (<45 seconds), which may aid forensic workflow efficiency.

We ascertain whether the sensitivity and fidelity of PL-Pb analysis can be exploited for shooting distance estimations. The first step for performing a shooting distance analysis is typically the measurement of a shooting distance series by shooting a firearm with ammunition of interest on a substrate at different distances (Figure 3A). Following this procedure, we create multiple shooting distance series ranging from 0 to 200 cm shooting distance (d) using a Glock 19 Gen5 pistol with 9 mm full metal jacket bullets (Figure 3A, see SI for details). We transfer the resulting GSR patterns to glass fiber cloths following the optimized transfer method, apply the PL-Pb reagent, and image the resulting PL pattern (Figure 3B). At $d = 0$ cm, we see a tightly clustered GSR pattern.[24] As we increase the shooting distance towards $d = 5$ cm, this cluster spreads out and we observe a polygonal pattern. At $d = 10$ cm we observe circular rippling patterns that are consistent with pressure waves from the muzzle blast. At 25 cm distance, we observe a pattern corresponding to the combustion plume. Beyond $d = 50$ cm, this pattern is not observable suggesting that the combustion plume is not reaching the substrate anymore (see SI), and we see the appearance of a speckling pattern 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. To test the reproducibility, we repeat the shooting series three times (see SI). We find similar pattern features at the same distances, hence showing that this distance measurement series gives reproducible results.

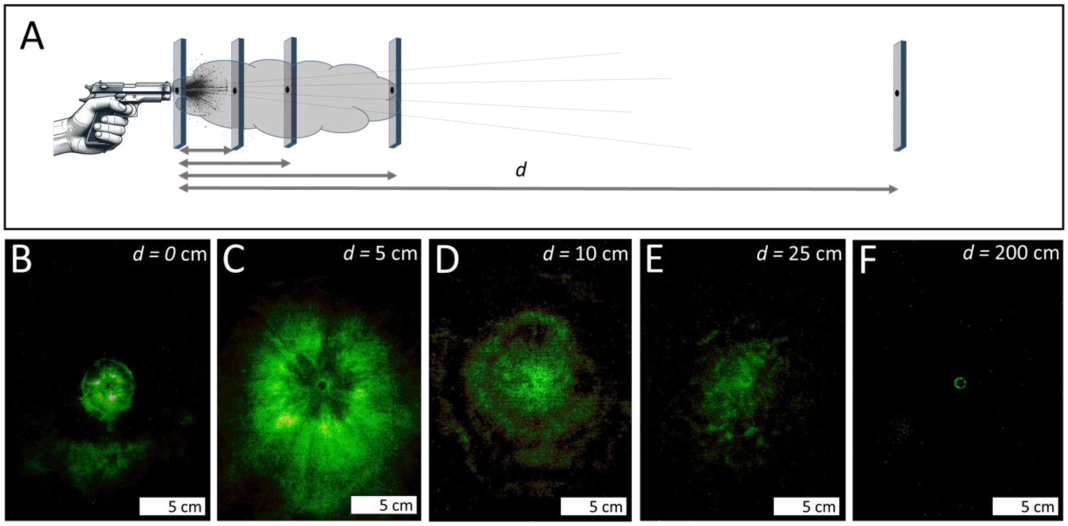

Figure 3: Shooting distance analysis using PL-Pb. (A) Setup for creating shooting distance series at distance *d*. Transferred GSR shooting distance patterns, showing for (B) *d* = 5 cm at tightly packed pattern, (C) *d* = 5 cm a polygonal pattern corresponding to the barrel rifling, (D) a rippling pattern of concentric rings consistent with the infrasonic compression waves, (E) *d* = 25 cm a central pattern from the combustion plume, and (F) *d* = 200 cm speckling pattern encompassing a bullet wipe. See SI for full series and reproductions.

We recognize that the sensitivity, robustness, instant results, and ability to perform mapping with refined spatial resolution make PL-Pb suitable for rapid presumptive testing of suspects and crime scene locations in a forensic shooting incident investigation. Motivated by these insights, we explore the potential of PL-Pb testing for assessing shooting incident involvement of potential suspects (Figure 4). For this, we use a modified version of the PL-Pb reagent that is non-toxic, and only mildly irritating to the skin, such that we can directly test hands of suspects. Upon spraying the hand of a shooter, bright PL is observable on the entire hand (Figure 4B). This instant and clearly visible GSR detection method opens unprecedented opportunities for rapid presumptive testing of shooting suspects.

Typically, washing hands makes GSR detection very difficult because these particles are often undetectable with EDS after a single wash[12,33]. Despite these complications, the ability to detect GSR can be relevant for forensic studies as suspects may try to tamper, conceal, or eliminate evidence. We therefore explore if the sensitivity of PL-Pb is sufficient to detect GSR after different washing protocols. Using the shooter's hands, we directly apply the PL-Pb reagent, resulting in a positive PL signal (Figure 4B). Even after extensive washing of the shooter's hands using different techniques, we still detect PL, likely because of the regular shooting activities of the shooter and the sensitivity of the PL-Pb test (See SI). To explore the sensitivity of the test, we analyze the hands of bystanders that are positioned ca. 2 meters to the side of the shooter. Before shooting, the hands of the bystanders test negative. However, after the shooter fires the pistol ten times, the hands of the bystanders emit clearly visible PL (Figure 4C). Casually washing of the hands of the bystanders and re-testing still yields a moderately heterogeneous PL signal (Figure 4D), and even after extensive washing and scrubbing of the hands we still detect weak heterogeneous PL signal that is focused on selected areas such as the fingernails and creases on the backside of fingers (Figure 4E). In contrast to previous reports using traditional GSR methods, with the PL-PB test Pb residues remain detectable even after attempts to remove the GSR. In a manner similar to the luminol test for blood, the presented method seems to be able to detect latent GSR traces.

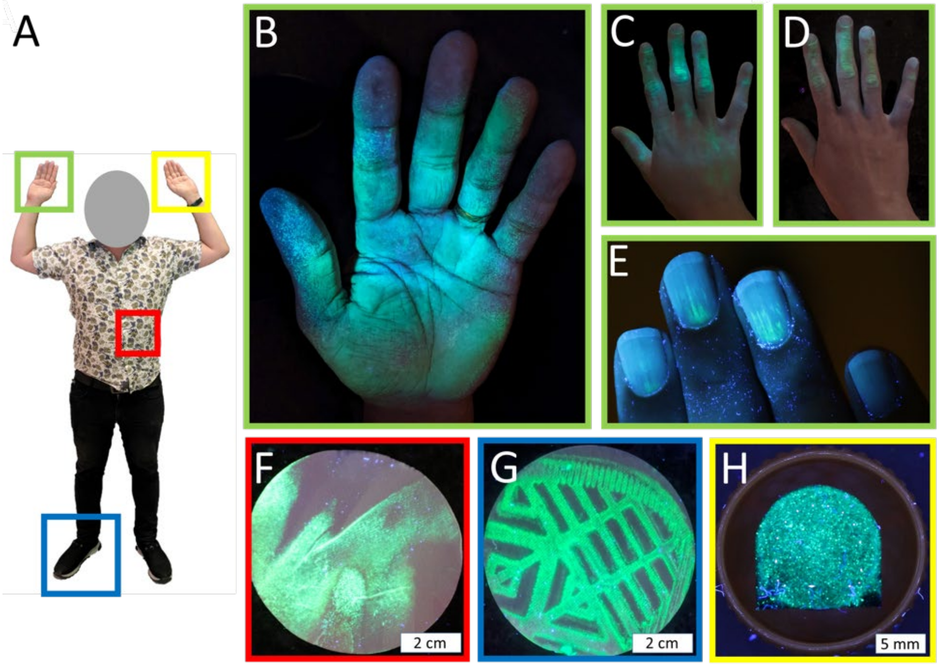

Figure 4: Assessing suspect involvement in a shooting incident using PL-Pb. (A) Marked areas for testing for GSR (sampling regions are color coded). (B) Direct PL-Pb testing on hand of shooter showing bright PL. (C) PL-Pb testing on hand of a bystander after shooting showing moderate PL, and (D) weak PL after washing with water and soap. (E) The hand of a bystander after thorough washing still showing a clear PL-Pb signal on the nails. (F) Indirect PL-Pb testing by wiping a glass fiber cloth over the clothing of a bystander showing bright PL. (G) Indirect testing of the shoe sole of a bystander. (H) SEM stub after stubbing shooter showing PL-Pb signal.

We find that GSR is detectable on shooters and bystanders by swiping hands, shoes, clothing, or other items on a crime scene (e.g. steering wheels from cars, pockets in jackets etc.) on a glass fiber swab and perform PL-Pb testing (Figure 4F, G). Although spatial location of GSR may be lost, the advantage of this indirect testing method is that potential irritation or discoloration by the corrosive reagent can be avoided which is desirable as the reagent is corrosive. Moreover, indirect testing limits potential interference with backgrounds (e.g. sweaty hands, fluorescent clothing) and other forensic methods, and GSR can be accumulated to achieve an even lower detection threshold.

To assess if PL-Pb is also compatible with well-established EDS analysis, we stub the hand of the shooter with a carbon tape used for EDS analysis. We apply the PL-Pb reagent and directly detect PL from lead particles on the stub (Figure 4H). Although compatibility with EDS analysis will require further research, these results suggest that PL-Pb testing can be valuable as pre-screening of shooting suspects. Hence, depending on the importance of sensitivity and spatial GSR information, either indirect or direct PL-Pb testing may be favorable, respectively.

Conclusion and Outlook

We here introduce a new GSR analysis technology based on the formation of lead halide perovskite semiconductors that emit bright light. We show that even trace amounts of lead in GSR are detected in relevant forensic scenarios by demonstrating direct detection of GSR and bullet wipes from full metal jacket bullets and combustion plumes. Moreover, we develop protocols for the transfer of GSR with high resolution and spatial fidelity. Using this transfer protocol, we design a shooting distance series which allows us to observe polygonal GSR patterns from barrel rifling, shock waves from the muzzle blast, and even submillimeter target features. We find that PL-Pb is particularly powerful for detecting trace amounts of lead. We exploit this sensitivity by testing shooters and bystanders and detect GSR on hands even after washing. In addition, instant testing of clothing, shoes, and other objects around a shooting incident is possible with PL-Pb and glass fiber swabs.

For practical forensic work, we foresee that our PL-Pb can be integrated easily. 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 our PL-Pb detection is highly compatible with current forensic workflows. In addition, we foresee that the fast transfer protocol and instant detection may speed up labor-intensive forensic practices and thus decrease this time-consuming bottleneck currently seen in GSR analysis.

The instant, robust and highly sensitive results of PL-Pb on the hands and clothing of shooters and bystanders opens new opportunities. Since the methylammonium bromide in the reagent is corrosive, we believe indirect testing using for instance swaps or cloth will be most attractive. First responders such as police officers can use the PL-Pb test to rapidly screen potential suspects and witnesses or define crime scenes to secure evidence. We note that detection of Pb itself is not conclusive for GSR. However, PL-Pb can be attractive as indicative test for prescreening of EDS stubs to speed up forensic work such that evidence is produced in time. Further research to establish the sensitivity of and compatibility between PL-Pb and EDS will therefore be highly relevant.

Further tuning of the testing method can be done for specific scenarios. For example, background fluorescence coming from clothing may hinder the interpretation of the signal. Currently, we overcome these complications by transferring GSR to a non-fluorescent substrate. However, we foresee that 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. Steps in these directions are currently being taken.

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 treads ranging from behavioral problems, aggression, and learning disabilities, to severe physical illnesses such as blindness, convulsions, cardiac diseases, and—ultimately—death.[34–37] 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] Already during this study, we found that our PL-Pb study works very well to identify lead in GSR at such locations. We therefore project that developing effective PL-Pb protocols can help to mitigate risks for lead poisoning from shooting ranges.

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.[3,4,38] Lead dust is a particular risk as it can be inhaled, 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.

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Competing interests

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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Table of contest

Caught green-handed: Lead in gunshot residue is detected by forming a perovskite semiconductor that emits bright green light. Lead dust patterns from bullet holes, combustion plumes, and polygonal riffling of the barrel are directly visualized. Instant presumptive testing of shooters and bystanders is demonstrated on hands, clothing, and shoes. Perovskite-based lead detection opens unprecedented opportunities for forensic investigations.