OSAC 2022-S-0036 Standard Method for the Chemical Testing of Copper and Lead from Suspected Projectile Impacts

Crime Scene Investigation & Reconstruction Subcommittee
Scene Examination Scientific Area Committee
Organization of Scientific Area Committees (OSAC) for Forensic Science

OSAC Organization of Scientific Area Committees for Forensic Science
Draft OSAC Proposed Standard

OSAC 2022-S-0036 Standard Method for the Chemical Testing of Copper and Lead from Suspected Projectile Impacts

Prepared by
Crime Scene Investigation & Reconstruction Subcommittee
Version 1.0
November 2022

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For more information about this important process, please visit our website at: https://www.nist.gov/topics/organization-scientific-area-committees-forensic-science/scientific-technical-review-panels.
Foreword

This standard is meant for scene investigators who are responsible for the documentation of a shooting scene and for shooting reconstructionists performing the on-scene documentation. It is recognized that some shooting scenes are processed and documented by scene investigators who will not be performing the final reconstructive analysis. However, their work is critical to any subsequent reconstructive efforts. This standard provides guidance for the chemical testing of suspected impact locations for projectile component transfer and the minimum documentation requirements for testing projectile impacts.

All hyperlinks and web addresses shown in this document are current as the publication date of this standard.

Keywords: Crime scene, shooting reconstruction, 2-nitroso-1-naphthol test, dithiooxamide test, sodium rhodizonate test, projectile impact, chemical testing
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Standard Method for the Chemical Testing of Copper and Lead from Suspected Projectile Impacts

1 Scope

This document provides scene investigators and scene reconstructionists with standard methods for the chemical testing of suspected projectile impacts for the presence of copper or lead residues. This standard is specifically meant to address the field processing of suspected projectile impacts. Collection of an item with a suspected projectile impact for testing in the controlled conditions of a laboratory should be considered when practical and warranted by the circumstances. Chemical processing and enhancement of gunshot residue patterns for the purpose of muzzle-to-target distance determinations or wound ballistics is beyond the scope of this standard.

2 Normative References

OSAC 2021-N-0019, Standard Practice for Documentation and Processing of Shooting Scenes

3 Terms and Definitions

For purposes of this document, the following definitions and acronyms apply.

3.1 2-nitroso-1-naphthol test
2-NN
A chemical test for the detection of copper.

3.2 bullet wipe
The discolored area around the periphery of a projectile entrance is caused by a physical transfer from the surface of a projectile to a target. This transfer can be composed of projectile lubricant, copper, lead, combustion products, bore debris, and/or projectile material. The word bullet in this term is inclusive of all projectiles.

3.3 control
Material of established origin is used to evaluate the performance of a test or comparison. Or a test performed to demonstrate that a test method works correctly and to ensure that data are valid. Positive controls confirm that the procedure will produce the expected result. Negative controls confirm that the procedure does not produce an unintended result. (ASTM E1732-12, OSAC Lexicon)

3.4 defect
A generic term for any surface damage. (OSAC proposed-Standard Practice for Documentation and Processing of Shooting Scenes)

3.5 dithiooxamide test
DTO
A chemical test for the detection of copper.

3.6 lead splash
A deposit made during a projectile impact is composed of a coating of lead particles too small to be individually distinguished without magnification. This deposit is observed adjacent to the projectile impact and is often deposited downrange from the impact location.

3.7 projectile
An object propelled with an initial velocity is then acted upon by gravity, air drag, and other outside forces. (OSAC proposed-Standard Practice for Documentation and Processing of Shooting Scenes)

3.8 projectile fragment
Any portion of a projectile that retains characteristics permitting it to be identified as having been part of a projectile. (OSAC proposed-Standard Practice for Documentation and Processing of Shooting Scenes)

3.9 projectile impact, n
Surface damage determined to have been caused by a projectile. (OSAC proposed-Standard Practice for Documentation and Processing of Shooting Scenes)

3.10 sodium rhodizonate test
NaRho
A chemical test for the detection of lead.

3.11 substrate control
A sample or test of an uncontaminated surface close to the area being tested that shares the same composition as the surface bearing the suspected evidence.

3.12 target, n
Any object struck by a projectile, regardless of whether it was struck intentionally. (OSAC proposed-Standard Practice for Documentation and Processing of Shooting Scenes)

4 Overview and Objective of Chemical Testing

This standard method is concerned with the testing for copper and lead residues, which are the most common constituents of manufactured projectiles such as lead bullets, copper-jacketed bullets, solid copper bullets, lead bullet cores, lead shot, lead shotgun slugs, and air gun pellets. In this standard, the meaning of the term projectile will be limited to those projectiles that have a copper or lead component.

Projectile impacts are generally identified based on their physical characteristics. However, in some cases, a lack of sufficient physical characteristics and scene context may not support a conclusion of whether a defect was caused by a projectile or some other mechanism. After appropriate documentation, chemical testing for projectile residues may offer additional data that can aid in making that determination.
Projecate residues are often present in or adjacent to a projectile impact due to the forceful interaction of the projectile with a target. This forceful interaction can abrade small amounts of metal or cause particles of metal to be ejected during impact and impinge on downrange surfaces. Sometimes this metal transfer is visible. There are chemical tests that can be used to enhance these residues or visualize latent residues.

The presence of some projectile residues can be used to yield other information about the projectile, such as its chemical composition or its direction of travel. Some projectile residues can also provide information about the firearm, such as its orientation relative to the residue deposition.

These tests require the practitioner to be able to differentiate the correct color changes for each reagent.

The chemical tests described below can be used to test for the presence of copper or lead from any source, but those uses are beyond the scope of this standard.

4.1 Considerations Before Chemical Testing

Chemical testing should be conducted prior to the insertion of trajectory rods into the projectile impacts being examined.

A shot fired from a relatively close distance from the target can deposit gunpowder particles or soot patterns on the target. If this is observed, a firearms examiner should be consulted about conducting a muzzle-to-target distance determination, and decisions regarding chemical testing should be deferred to the firearms examiner to ensure proper preservation of evidence. This could include chemical tests beyond the scope of this standard.

4.2 Order of Testing

If testing the impact site for both copper and lead, the order of examination is critical. The sodium rhodizonate test for lead will interfere with the two tests for copper residues. Therefore, when testing for both copper and lead, the copper test(s) shall be conducted before the lead test. Sections 5 and 6 describe detailed materials and methods for conducting chemical tests for the presence of copper and lead residues, respectively.

When more than one test is used, the documentation of the testing shall clearly describe the order in which they were conducted.

4.3 Control Testing

The results of all control tests shall be documented in the notes.

4.3.1 Substrate Controls

If pre-existing copper or lead is suspected on the target surface, it shall be evaluated. Examples of potentially problematic target surfaces include brass doorknobs (containing copper) or surfaces covered in lead-based paint. While not always required, a substrate control may be useful to determine if the background material is causing a positive reaction.

A substrate control test conducted away from any potential projectile impacts may be performed when the practitioner suspects background interference/contamination.

4.3.2 Reagent Controls
To ensure that the reagents produce reliable results, the use of positive and negative controls is necessary. Reagents shall be tested with positive and negative controls at the time the reagents are prepared and just prior to each test session.

A common practice for controls is to mark a piece of clean filter paper with a known source of copper or lead. The mark is a positive control and should generate the expected color reaction. The surrounding unmarked area is the negative control and should not generate a color reaction. Reagents that do not pass the positive and negative control tests shall not be used.

4.4 Recording Reagent Lot Numbers

Any chemical reagents or precursors used in their preparation that is used on evidence or at a scene shall be traceable to the specific manufacturer and lot number used.

4.5 Chemical Safety

Safe handling and preparation of chemical reagents are beyond the scope of this standard. Personnel preparing or using these reagents shall understand basic safety when working with these chemicals and review their safety data sheets (SDS) prior to their use. When spraying reagents, respiratory protection or proper ventilation shall be used.

5 Chemical Testing for Copper Residues

5.1 2-Nitroso-1-Naphthol (2-NN) and Dithiooxamide (DTO) Tests

5.1.1 Overview and Theory

When a copper-bearing projectile impacts a surface, trace residual copper may be transferred at the contact point. There are two different chemical tests for copper: 2-nitroso-1-naphthol (2-NN) and dithiooxamide (DTO). The practitioner may choose which of these two tests to use. Because each reagent has its own color reaction (pink for 2-NN and dark green for DTO), consideration of the color of the substrate can help the examiner decide which reagent to use. The copper test selected should be whichever one provides the best color contrast against the substrate background color.

Prepared solutions of 2-NN and DTO have a limited shelf life and should be prepared at the time of use. Reagents prepared in advance may be used, but the reactivity of any reagent shall be confirmed with controls prior to use.

See Appendices B and C for recommended materials and reagent preparation.

5.2 Protocol for Copper Testing of Suspected Projectile Impacts

5.2.1 Pre-testing procedures

All prepared solutions shall be control tested, both at the time the reagents are prepared and just prior to each test session. If loss of potency is suspected, additional control tests may be appropriate.

The results shall be recorded in the case notes. Visual assessments, notes, and photographs of the suspected impact shall be conducted prior to the chemical testing. Any potentially probative biological or trace evidence shall be collected prior to chemical testing. Trajectory analysis tools shall not be inserted into a defect prior to chemical testing. Any prior physical contact with the defect shall be documented and considered as a potential source of contamination.
5.2.2 Transfer/Lift Method

a) Cut a section of filter paper larger than the area of interest.

b) Moisten the filter paper with the 2:5 ammonium hydroxide solution.

c) Without rubbing, firmly press and hold the moistened filter paper onto the surface to be examined. Ensure that the filter paper is in contact with the entire area of interest (e.g., bent car metal with a suspected impact that has caused a deep defect).

d) After placing reference marks to record the orientation on the filter paper, remove it, and examine for any color transfer that could mask the color reaction of 2-NN or DTO. Choose the best reagent to visualize the reaction. It is recommended to use a pencil for the reference marks to avoid bleeding of the marks when reagents are applied.

e) Apply the 2-NN or DTO solution onto the filter paper.

i) A positive result for copper with the 2-NN solution will result in an immediate pink color change.

ii) A positive result for copper with the DTO solution will result in an immediate dark green color reaction.

f) The results shall be photographed in color with a scale.

i) In some instances, the shape and position of a positive reaction can be useful for a shooting reconstructionist.

5.2.3 Direct Application Method

If the area of interest is on a relatively porous material and the color responses of the reagents can be visualized over the background colors, lightly apply the reagents directly to the area in the same order as described above in 5.3.2. Applying an excessive volume of liquid reagents during the direct application method is a common error because it can dilute or wash away the color reaction. The transfer/lift method can allow better visualization when the background color masks the reaction color. One can always make a direct application after the lifting technique has been attempted. Prior to applying the reagents, the area of interest shall be examined and photographed to identify pre-existing background colors. The direct application technique should not be used routinely in the field on items such as clothing that are better suited for analysis in the laboratory.

5.2.4 Documentation of Results

Positive reactions shall be documented with photography using a scale and notes.

Negative reactions shall be noted and may be photographed.

6 Chemical Testing for Lead Residues

6.1 Sodium Rhodizonate Test

6.1.1 Overview and Theory

When a projectile, whether jacketed or not, impacts a target surface, it can leave trace amounts of lead at or around an impact site. By partially dissolving lead in an acidic buffer (such as pH 2.8 tartrate or 15% acetic acid), the impact site can be tested with sodium rhodizonate solution. (Note: Other validated acidic buffers can also work in this reaction and may be substituted if agency protocols allow.) Upon application of the sodium rhodizonate reagent, a pink color change will occur when a lead is present. A follow-up test with 5% hydrochloric acid (HCl) will change a positive pink color to purple-blue color, further supporting the presence of lead. The tartrate buffers solution and dilute HCl solution may be stored for extended
periods of time at room temperature, in an airtight container, and out of strong light. The sodium rhodizonate solution should be made, and control tested just prior to use. Reagents prepared in advance may be used, but their reactivity shall first be confirmed with controls.

See Appendices B and C for recommended materials and reagent preparation.

6.2 Protocol for Lead Testing of Suspected Projectile Impacts

6.2.1 Pre-testing procedures

All prepared solutions shall be control tested, both at the time the reagents are prepared and just prior to each test session. If loss of potency is suspected, additional control tests may be appropriate. The results shall be recorded in the case notes. Visual assessments, notes, and photographs of the suspected impact shall be conducted prior to the chemical testing. Any potentially probative biological or trace evidence shall be collected prior to chemical testing. Trajectory analysis tools shall not be inserted into a defect prior to chemical testing. Any prior physical contact with the defect shall be documented and considered as a potential source of contamination.

6.2.2 Transfer / Lift Method

The following test method shall be followed with any deviations documented in the notes:

a) Cut a section of filter paper larger than the area of interest.
b) Moisten the filter paper with tartrate buffer solution. Another validated acidic buffer may be substituted if agency protocols allow.
c) Without rubbing, firmly press and hold the moistened filter paper onto the surface to be tested. Ensure that the paper is in contact with the entire area of interest (e.g., bent car metal with a suspected impact that has caused a deep defect).
d) After placing any reference or orientation marks on the paper, remove it and examine for any color transfer.
e) Apply sodium rhodizonate solution onto the filter paper
   i) A positive result for lead will produce an immediate pink color change.
f) To neutralize the background color from sodium rhodizonate application, the 5% HCl application may be used. A purple-blue color change further supports the presence of lead.
g) To neutralize the background color from sodium rhodizonate application, the 5% HCl application may be used. A purple-blue color change further supports the presence of lead.
h) The results shall be photographed in color with a scale.
   i) In some instances, the shape and position of a positive reaction can be useful for a shooting reconstructionist.

6.2.3 Direct Application Method

If the area of interest is on a relatively porous material and the responses of the reagents can be visualized over the background colors, the direct application method can be used to lightly spray the reagents directly onto the suspected impact area in the same order as described above in 6.3.2.

Spraying an excessive volume of liquid reagents during the direct application method can dilute or wash away the color reaction. The transfer/lift method can allow better visualization when the background color masks the reaction color. The direct application method may be performed after the lifting method is attempted. Prior to applying the reagents, the area of interest shall be examined and photographed to
identify pre-existing background colors. The direct spray technique should not be used routinely in the field on items such as clothing that are better suited for analysis in the laboratory.

6.2.4 Documentation of Results

The positive color reactions can fade quickly. Documenting the reaction shall occur immediately after the application of the sodium rhodizonate and again if the HCl overspray step is conducted. Documentation shall include color photography with a scale that can also be supplemented with notes and sketches.

Negative reactions shall be documented and may be photographed.

7 Commercial Projectile Impact Test Kits

A variety of commercially available kits are marketed specifically for the testing of suspected projectile impact locations for the detection of copper and lead residues. Such kits may be used if the instructions are followed. Prior to use at scenes, appropriate positive and negative controls shall be tested and documented. If the contents of the pre-packaged commercial kit are unclear, the scene investigator should know which chemical reagent is in the kits they use. If not expressly described in the kit instructions, this information might be present in the safety data sheets (SDS) for the product.

7.1 Documentation of Results

Positive reactions shall be documented with photography using a scale and notes.

Negative reactions shall be noted and may be photographed.

8 Limitations

8.1 Practical Limitations

8.1.1 Projectile residues are regularly but not always transferred during impact. For that reason, negative results must be carefully interpreted.

8.1.1.1 When projectiles impact materials, the substance that is physically contacted can be removed (e.g., glass, asphalt). This can yield a negative result, even if the defect was actually a projectile impact.

8.1.1.2 While lead and copper are the most commonly encountered projectile metals, in rare instances, projectiles can be made of other materials. This can yield a negative result, even if the defect was actually a projectile impact. Therefore, if the scene evidence suggests that an uncommon projectile metal may have been used, negative results for copper or lead should be interpreted with caution.

8.1.1.3 Manufactured projectiles that are not made of copper or lead can still be contaminated with those elements from primer residues and bore fouling. Therefore, those elements can be transferred to an impact and yield a positive test result for copper or lead.

8.1.2 The target material could contain copper or lead that would produce a positive reaction.

8.1.3 The trace amounts of projectile residues transferred at a projectile impact site can be further reduced by environmental factors or poor handling, resulting in mechanical loss of those projectile residues.
8.1.4 The wide variety of target materials and projectile types has a greater influence on the results than the detection limits of the reagents. Because projectile impacts are typically high-energy events, the expected transfers generally exceed the detection limits of the reagents.

8.1.5 The reagents can degrade over time.

8.2 Interferents

8.3.1 Physical interferents that can mask the results of chemical testing at projectile impacts include target color, target cleanliness, and the presence of biological materials.

8.3.2 The chemical makeup of the target material should be considered as it can prevent or detract from the reagent's effectiveness. For example, alkaline substances like concrete blocks can neutralize acidic solvents such as acetic and tartaric acids, thereby influencing the test results.

9 Results and Interpretation

9.1 Chemical Test Results

9.1.1 A positive reaction for copper and observed physical characteristics on a suspected projectile impact using either the 2-NN or DTO test supports the conclusion of an impact from a source of copper.

9.1.2 A positive reaction for lead and observed physical characteristics on a suspected projectile impact using the sodium rhodizonate test supports the conclusion of an impact from a source of lead.

9.1.3 A negative result for either test means that copper or lead was not detected.

9.2 Interpretation

9.2.1 Positive results are supportive of the opinion that a defect was caused by a projectile impact when other physical characteristics, location, and scene context are present.

9.2.2 Negative results, due in part to limitations as discussed above in section 8, do not necessarily eliminate a defect as having been caused by a projectile impact and shall be interpreted considering physical characteristics, location, and scene context.
Annex A
(informative)

Materials

The following materials are needed to perform the chemical tests for copper and lead.

a) Small sprayers or dropper bottles
b) Sheets of clean, white filter paper
c) Latex or nitrile gloves
d) Eye protection
e) Copper source for positive control
f) Lead source for positive control
g) Graduated cylinder/beaker
h) Scale/Balance
i) Reagents/chemicals
   i) 2-nitroso-1-naphthol (2-NN)
   ii) dithiooxamide
   iii) 95-100% ethanol
   iv) 28-30% ammonium hydroxide (NH₄OH)
   v) sodium bitartrate
   vi) tartaric acid (powdered)
   vii) glacial acetic acid
   viii) distilled water
   ix) 37% hydrochloric acid (HCl)
   x) sodium rhodizonate (powder)
Annex B
(informative)

Reagent Preparation

The following list of materials and mixing instructions are used for reagent preparation.

B.1 2-NN solution

B.1 0.2 g of 2-nitroso-1-naphthol in 100 ml of ethanol

Pour 0.2 grams of the dry powder of 2-nitroso-1-naphthol into a beaker and add 100 milliliters of ethanol. Swirl the container to allow the powder to dissolve. The reagent will be a dark color after mixing. Transfer the liquid reagent into the appropriate spray bottle or dropper.

B.2 DTO solution

0.2 g of dithiooxamide in 100 ml of ethanol

Pour 0.2 grams of the dry powder of dithiooxamide into a beaker and add 100 milliliters of ethanol. Swirl the container to allow the powder to dissolve. The reagent will be a dark color after mixing. Transfer the liquid reagent into the appropriate spray bottle or dropper.

B.3 2:5 dilution factor of ammonium hydroxide

20 ml of ammonium hydroxide (NH₄OH) diluted to a total of 50 ml with distilled water

To obtain the 2:5 dilution factor, measure 30 ml of distilled water and slowly add 20 ml of concentrated ammonium hydroxide (NH₄OH). Transfer the liquid reagent into the appropriate spray bottle or dropper. Ammonium hydroxide is a strong base, and the concentrated liquid can cause chemical burns. Always use appropriate personal protective equipment when mixing chemicals.

B.4 pH 2.8 tartrate buffer solution

1.9 g of sodium bitartrate and 1.5 g of tartaric acid in 100 ml of distilled water

Weigh out the powder sodium bitartrate and tartaric acid and combine in a glass container. Add 100 ml of warm distilled water and stir until the powder has completely dissolved. Transfer the liquid reagent into the appropriate spray bottle or dropper.

B.5 15% acetic acid

15 ml glacial acetic acid in 85 ml distilled water

Measure 85 ml of distilled water and slowly add 15 ml of glacial acetic acid. Transfer the liquid reagent into the appropriate spray bottle or dropper. Glacial acetic acid is a strong acid, and the concentrated liquid can cause chemical burns. Always use appropriate personal protective equipment when mixing chemicals.

B.6 0.2% sodium rhodizonate solution
0.2 g of sodium rhodizonate in 100 ml of distilled water

Sodium rhodizonate powder is added to distilled water until saturated, which will be apparent when, after stirring, a small amount of undissolved powder remains in the bottom of the container. Transfer the liquid reagent into the appropriate spray bottle or dropper.

**B.7 Dilute (5%) hydrochloric acid (HCl) for follow-up testing**

5 ml concentrated HCl diluted to 100 ml with distilled water

Measure 100 ml of distilled water and slowly add the 5 ml of concentrated hydrochloric acid (HCl). Transfer the liquid reagent into the appropriate spray bottle or dropper. Concentrated hydrochloric acid is a strong acid, and the concentrated liquid can cause chemical burns. Always use appropriate personal protective equipment when mixing chemicals.
Annex C
(informative)

Bibliography

This is not meant to be an all-inclusive list, as the group recognizes other publications on this subject may exist. At the time this document was drafted, these were the publications available for reference. Additionally, any mention of a particular software tool or vendor as part of this bibliography is purely incidental, and any inclusion does not imply endorsement.


