FBI Laboratory
Firearms/Toolmarks Unit (FTU)
The Sodium Rhodizonate Test for Lead Residues

1 Scope

The Sodium Rhodizonate Test is designed for detecting depositions of vaporous and particulate lead around a suspected bullet hole as a basis for estimating muzzle-to-target distances. Such depositions may be on evidence items such as clothing, furniture, bedding, and wallboard.

2 Equipment/Materials/Reagents

Electronic balance (minimum weighing range must exceed 0-50 grams, but not exceed 0-3000 grams; the linearity specification must not exceed 0.1 gram); Microspatula; Filter paper; Aerosol spray equipment; Blotters/brown wrapping paper; Glass stirring rod; Disposable PVC gloves; Hot plate/magnetic stirrer; Polyethylene bottles; Laboratory coat; Glass beaker (500 ml); Exhaust hood; Concentrated hydrochloric acid (reagent grade or better); Sodium bitartrate (reagent grade or better); Tartaric acid (reagent grade or better); Sodium rhodizonate (reagent grade or better); De-ionized and/or distilled water; Glacial acetic acid (reagent grade or better).

3 Standards and Controls

A lead bullet is used as a positive control for the Sodium Rhodizonate Test (SoRho). If a chemical reagent must be prepared before an examination, the following information must be recorded on the appropriate FTU Chemistry Log which can be found in the Firearms/Toolmarks Unit, *Comprehensive Gunshot Residue Examinations in Muzzle-to-Target Distance Determination* procedure: chemical produced, preparer, date (lot number), parent chemical, FBI Laboratory serial number and the performance check. The chemical lot number for reagents used during examinations will be recorded in the examination notes.

4 Calibration

Not Applicable.

5 Sampling

Not Applicable.

6 Procedures

6.1 Preparation of Reagents and Test Media

6.1.1 Storage of prepared chemicals and test media should be such that contamination is not possible. Storage containers should be kept sealed until the contents are needed. Fractions or multiples of the weights and volumes indicated may be used as appropriate to the amount of work to be done. For disposal of the chemicals used for this procedure, refer to the Guideline for Hazardous Waste Management in the Firearms-Toolmarks Unit (FTU), copies of which are maintained in the Chemistry Room.

6.1.2 Preparation of the Sodium Rhodizonate Solution

Place a small amount of sodium rhodizonate in a small beaker and add sufficient de-ionized and/or distilled water to make a saturated solution approximately the color of strong tea. The solution is saturated if slight sediment is noted on the bottom of the beaker after stirring with a clean glass stirring rod. Make only enough solution for immediate use.

6.1.3 Preparation of 2.8 pH Buffer Solution

6.1.3.1 Dissolve 1.9 grams of sodium bitartrate and 1.5 grams of tartaric acid per 100 milliliters of distilled water. This usually requires both heat and agitation. A combination hot plate/magnetic stirrer can be used for this.

6.1.3.2 Store the solution in a properly labeled and sealed container (the latter to prevent clouding). There is no known limit to the shelf life of this reagent.

6.1.4 Preparation of the Dilute (5%) Hydrochloric Acid Solution

6.1.4.1 Combine 5 milliliters of concentrated acid with 95 milliliters of distilled water. Pour the acid into the water to preclude potential spattering of undiluted acid.

6.1.4.2 Store the solution in a properly labeled and sealed container. Solution can be stored indefinitely.

6.1.5 Preparation of a 15% Acetic Acid Solution

6.1.5.1 Combine 150 milliliters of glacial acetic acid with 850 milliliters of distilled water. Gently pour the acid into the water to preclude the potential spattering of undiluted acid.

6.1.5.2 Store the solution in a properly sealed and labeled container. There is no known limit to the shelf life of this reagent.

6.1.6 Preparation of Controls

6.1.6.1 Positive Control

A lead bullet from the FTU ammunition room will be wiped across a piece of test material and the material then processed for the expected blue-violet reaction. The results will be recorded in the examiner's notes.

6.1.6.2 Negative Control

Observing the absence of any blue-violet color development on the non-wiped portions of the test material is sufficient for a negative control. The results will be recorded in the examiner's notes.

6.2 Direct Application to Light-Colored Items of Evidence

6.2.1 Spray the appropriate area of the questioned item with the previously prepared saturated solution of sodium rhodizonate.

6.2.2 Spray the same area of the questioned item with the previously prepared tartaric acid/sodium bitartrate buffer solution. This solution will eliminate the general yellow background color caused by the sodium rhodizonate and will establish a local pH of 2.8, turning any lead, as well as other metals that may be present, a pink color.

6.2.3 Spray the same area with the previously prepared dilute hydrochloric acid solution. The presence of lead is specifically determined wherever the previous pink color fades out and leaves a blue-violet color in its place; this indicates lead and only lead. Be very aware that a positive (blue-violet) result may abruptly fade. Note the results immediately after applying the dilute hydrochloric acid solution.

6.3 The Reverse Transfer Method for Dark-Colored Items that Would Mask the Blue-Violet Coloration of a Positive Test Result

6.3.1 Place a piece of filter paper over the appropriate area of the questioned item.

6.3.2 Index the filter paper relative to the garment or other item to indicate the location of such things as suspected bullet holes, seams, buttons, button holes, pockets, rips, and tears. Indexing in pencil is preferable since ink may bleed during the application of reagents.

6.3.3 Uniformly dampen the filter paper on the questioned item by spraying with a 15% solution of glacial acetic acid.

6.3.4 Cover the dampened filter paper with several layers of dry filter paper. Apply a hot iron to the filter paper and iron until the paper is dry.

6.3.5 Remove the filter paper which was in direct contact with the evidence item, and process

it using the steps in 6.2 above. Note that any positive (blue-violet) indications are a mirror image of the deposition on the questioned item.

6.3.6 Prompt note-taking is essential in that sometimes the color can fade rapidly and unpredictably. When dry, filter paper exhibiting positive results should be properly marked in ink for future identification, and retained in the case file or returned to the contributor. Filter paper exhibiting no results can be discarded.

6.4 The Standard Transfer Method, Normally a Last Resort

6.4.1 Process the questioned item by following all steps in 6.2 above.

6.4.2 Blot the appropriate area of the questioned item using untreated filter paper.

6.4.3 Note any positive results. Such transfers usually reflect positive results which are very vague and indistinct in form. See 6.3.6 for proper handling.

7 Calculations

Not Applicable.

8 Uncertainty of Measurement

Not Applicable.

9 Limitations

The Sodium Rhodizonate and Modified Sodium Rhodizonate Tests yield results for the presence of lead regardless of whether these are related to the discharge of a firearm or the passage of a bullet.

10 Safety

Since many of the procedures involve the spraying of reagents in an aerosol form. All spraying should be done in an exhaust hood that has an air flow velocity of 60-120 feet/minute. Protective latex or vinyl gloves will be worn at all times.

11 References

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Dillon, J.H., "A Protocol for Gunshot Residue Examination in Muzzle-to-Target Distance Determinations," AFTE Journal, Vol. 22, No. 3, 1990, pp. 257-274.

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Fiegel, F. and Anger, V., Spot Tests in Inorganic Analysis, 6th ed, Elsevier Publishing Co, New York, 1972.

Lekstrom, A.J. and Koons, R.D., "Copper and Nickel Detection on Gunshot Targets by Dithiooxamide Test," Journal of Forensic Sciences, Vol. 31, No. 4, 1986, pp. 1283-1291.

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FBI Laboratory Quality Assurance Manual

FBI Laboratory Operations Manual

FBI Laboratory Safety Manual

FBI Laboratory, FTU Quality Assurance Manual

Rev. #	Issue Date	History
0	07/10/06	Original issue for ASCLD/LAB-*International* accreditation
1	02/17/07	Updated section 3 including use of FTU Chemistry Log (Appendix C)
2	11/05/07	Potassium Chloride (KCl) deleted from section 2. Deleted section 6.1.6 and renumbered section, and sections 6.5 and 6.6 were deleted due use of KCl. See DTO procedure sections 6.2 and 6.3 for method. Removed reference to standards in section 3. Updated section 4 by removing calibration information.
3	08/21/12	Section 3 added cite where Chemistry Logs can be found. Section 6.1.2 added de-ionized and/or distilled water.

Approval

Redacted - Signatures on File