Designation: E3329 − 21

An American National Standard

Standard Practice for Establishing an Examination Scheme for Explosive Residues[1]

This standard is issued under the fixed designation E3329; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers the evaluation, selection, and application of techniques to establish an examination scheme for use by forensic explosives examiners to identify residues from low and high explosives. A foundation for the consistent approach to the analysis of visible and non-visible explosive residues from post-blast or other explosive-related scenes is provided. Methods for the development of identifying information that follows an efficient order of testing are described.

1.2 This practice establishes requirements for the use of visual, physical, analytical, and instrumental techniques that provide structural and chemical information for an identification of an explosive residue.

1.3 Techniques used in the examination of explosive residues include visual and microscopical inspection, physical characterization, ignition susceptibility testing, chemical and spot testing, and instrumental methods.

1.4 This standard is intended for use by competent forensic science practitioners with the requisite formal education, discipline-specific training (see Practice E2917), and demonstrated proficiency to perform forensic casework (refer to the T/SWGFEX Suggested Guide for Explosives Analysis Training).

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E620 Practice for Reporting Opinions of Scientific or Technical Experts

E860 Practice for Examining And Preparing Items That Are Or May Become Involved In Criminal or Civil Litigation

E1732 Terminology Relating to Forensic Science

E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs

E2998 Practice for Characterization and Classification of Smokeless Powder

E2999 Test Method for Analysis of Organic Compounds in Smokeless Powder by Gas Chromatography-Mass Spectrometry and Fourier Transform Infrared Spectroscopy

E3255 Practice for Quality Assurance of Forensic Science Service Providers Performing Forensic Chemical Analysis

E3196 Terminology Relating to the Examination of Explosives

E3253 Practice for Establishing an Examination Scheme for Intact Explosives

2.2 *Other Resources:*

Technical/Scientific Working Group for Fire and Explosion Analysis (T/SWGFEX) Suggested Guide for Explosive Analysis Training[3]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, see Terminology E1732, Practice E3253, and Terminology E3196.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *explosive residue, n*—material remaining from an explosive deflagration or detonation or from direct contact with an explosive. Explosive residue generally does not have a

[1] This practice is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics.

Current edition approved Nov. 1, 2021. Published January 2022. DOI: 10.1520/E3329-21.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, https://www.nist.gov/system/files/documents/2018/09/21/twgfex_suggest_guide_for_explosive_analysis_training.pdf.

discernible morphology, can be visible or non-visible, and can contain uninitiated and post-combustion components.

3.2.1.1 *Discussion*—Visible explosive residue can be present in large (grams) and small (single particle or single crystal) quantities.

4. Quality Assurance

4.1 The processing of residue evidence samples should be separated in space from other intact explosives to prevent incidental contamination. If space does not allow for this, then process intact explosives and residue evidence separately by time. Ensure appropriate precautions are in place to prevent contamination (**1-7**).[4]

5. Summary of Practice

5.1 Multiple techniques and methods are used in the identification of explosive residues.

5.2 Examination of suspected explosive residues starts with macroscopical and microscopical observations. The interpretation of these initial tests requires that the examiner has a working knowledge of explosives. While characteristics observed by visual examination results can indicate an explosive residue, it is necessary to use additional analytical techniques to identify suspected explosive residues. Refer to Practice E3253 if suspected intact explosives are recovered.

6. Significance and Use

6.1 This practice is designed to assist the forensic explosives examiner in selecting and organizing an analytical scheme for identifying explosive residues. The amount and condition of the sample, as well as the availability of instrumental techniques, will determine the selected analytical scheme.

6.2 The forensic explosives examiner considers relevant issues about the case investigation and submitted items, such as sample size, complexity and condition, environmental effects, and collection methods used. Considerations include test methods, sample preparation schemes, test sequences, and acceptable degrees of sample alteration and consumption that will be different for each case submission.

6.3 This practice is used when insufficient material is present to follow Practice E3253.

6.4 This practice is used in conjunction with the referenced documents.

6.5 This practice does not attempt to address all the issues regarding sample analyses. There could be additional tests or analyses performed to provide further discrimination and characterization of samples.

7. Selection of Appropriate Analytical Techniques

7.1 Refer to Practice E3253 for information on the variety of techniques that can be used.

7.1.1 Some of these techniques can irreversibly alter the sample. For example, water extractions can result in ion exchange. A portion of the original sample should be preserved for potential future examination; however, preservation will depend on the submitted sample size, sample preparation, and analytical techniques used.

7.2 A scheme for examining explosive residues is outlined in Fig. 1.

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.

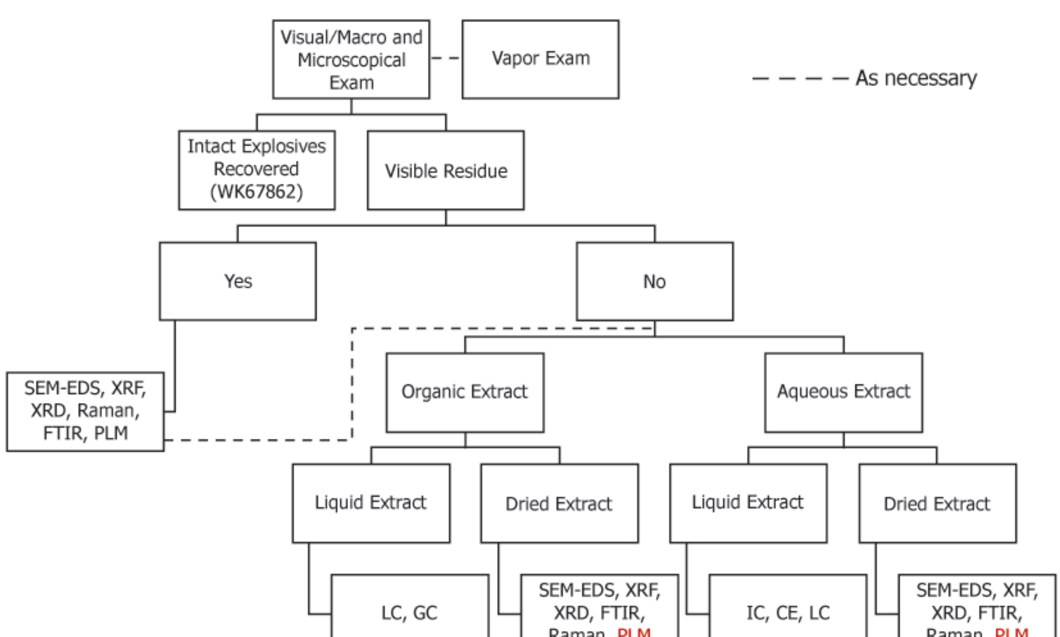

FIG. 1 Scheme for Explosive Residue Examinations

7.2.1 Residue quantities greater than approximately 1 gram can be initially analyzed following Practice E3253, regardless of whether the material is unexploded, initiated, or a mixture of both.

7.2.2 If limited sample is available (less than approximately 1 gram), use an analytical scheme that yields the most information and consumes the least amount of sample. Fig. 1 does not imply that other examinations should be excluded or that the order of the procedures in the figure is predetermined.

7.3 *Visual/Macro and Microscopical Exams:*

7.3.1 Begin with a review of the information submitted with the evidence, including the condition of the packaging and labeling, and document any potential for cross-contamination between samples.

7.3.1.1 Record any potential for cross-contamination or discrepancies and take this into account in the final evaluation of the analytical data.

7.3.2 Conduct a visual and stereo microscopical assessment of each item, prior to (when the packaging permits), and after opening the evidence packaging. Provide a written description of the general condition, size, shape, and color of each sample and photograph when possible.

7.3.2.1 This preliminary assessment provides information for the selection of an analytical scheme and can identify potential complexities in the review of analytical data obtained.

7.3.2.2 Prior to analysis, consider the potential effect on other examinations, such as trace, latent prints, and DNA (**8-10**).

7.3.2.3 If items are submitted in suitable containers (for example, paint cans, glass jars, or nylon bags), a vapor examination can be performed to identify certain organic explosives prior to conducting a full visual assessment. (**Warning—**Perform this analysis in a timely manner as vapors can dissipate rapidly.)

7.3.2.4 Examine visible material to determine if it is visually similar to an explosive, related to an explosive, non-explosive or reaction products of an explosive.

7.3.2.5 Follow Practice E3253 for recovered explosive materials with a discernible morphology or a quantity greater than approximately 1 gram. Further residue analysis could still be required for some materials.

7.3.2.6 Utilize a solvent extraction scheme to isolate residues of explosives, explosive components or reaction products, if no visible material is observed or material cannot be physically removed (**11-13**).

NOTE 1—The choice of solvent can be influenced by the damage to the evidence and other information related to the scene, which can indicate the type of explosive involved.

8. Analytical Requirements for Identification

8.1 Analysis and identification requirements for residues are dependent on a variety of factors, such as the type of explosive used, evidence collected, packaging, environmental conditions (**14-17**), and instrumental techniques available.

8.1.1 Explosive residue analysis includes the identification of original components or reaction products, or both (**18**).

8.2 *Inorganic Low Explosives:*

8.2.1 Inorganic low explosives include commercially manufactured products, such as black powder and black powder substitutes, and solid oxidizer and fuel mixtures.

8.2.2 Original components or reaction products of inorganic low explosives are identified by a variety of techniques. For identification requirements of original components refer to Practice E3253.

8.2.2.1 If visible residues are present, note their physical characteristics.

8.2.2.2 If visible residues can be physically removed, analyze using one of the following: X-ray fluorescence (XRF), scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS), or chemical spot test; and one of the following: X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Raman, or polarized light microscopy (PLM).

8.2.2.3 If no visible residues are present, or if techniques listed in 8.2.2.2 do not yield adequate results, analyze extracts (filtered or concentrated as needed) using ion chromatography (IC), capillary electrophoresis (CE), mass spectrometry (MS), gas chromatography (GC), or liquid chromatography (LC) with appropriate confirmation techniques, if necessary. Extracts can also be dried and analyzed using one of the following: XRF, SEM-EDS, or chemical spot test; and one of the following: XRD, FTIR, Raman, or PLM.

NOTE 2—Chemical spot tests are destructive and should only be conducted if sufficient residue material can be physically removed, collected, and preserved for further analysis.

8.2.3 *Black Powder:*

8.2.3.1 Black powder is composed of potassium nitrate, sulfur and charcoal.

8.2.3.2 Reaction products of black powder can include potassium sulfate, potassium carbonate, potassium sulfide, potassium thiocyanate, potassium bicarbonate, and potassium nitrite.

8.2.4 *Pyrodex:* [5]

8.2.4.1 Pyrodex is composed of potassium nitrate, potassium perchlorate, sulfur, sodium benzoate, cyanoguanidine, and charcoal.

8.2.4.2 Reaction products of Pyrodex can include potassium sulfate, potassium chloride, potassium carbonate, potassium sulfide, potassium thiocyanate, potassium bicarbonate, potassium nitrite, and potassium chlorate.

8.2.5 *Triple Seven* [6]

8.2.5.1 Triple Seven is composed of potassium nitrate, potassium perchlorate, sodium benzoate, cyanoguanidine, sodium 3-nitrobenzoate and charcoal.

8.2.5.2 Reaction products of Triple Seven can include potassium chloride, potassium carbonate, potassium bicarbonate, potassium nitrite, and potassium chlorate.

8.2.6 *Ascorbic Acid-Based Black Powder Substitutes:*

8.2.6.1 The original components include ascorbic acid and at least potassium nitrate or potassium perchlorate.

8.2.6.2 Reaction products can include potassium carbonate, potassium chloride, potassium nitrite, potassium chlorate, and

[5] Pyrodex is a trademark of Hodgdon Powder Company, Inc., Shawnee, KS.

[6] Triple Seven is a trademark of Hodgdon Powder Company, Inc., Shawnee, KS.

ascorbic acid degradation products (for example, threonic acid, monohydrated diketogulonic acid, and oxalic acid).

8.2.7 *Solid Oxidizer and Fuel Mixtures:*

8.2.7.1 Examples of solid oxidizers include potassium perchlorate, potassium chlorate, ammonium perchlorate, ammonium nitrate, potassium nitrate, sodium nitrate, barium nitrate, strontium nitrate, potassium permanganate, and iron oxide. Examples of solid fuels include aluminum, magnesium, magnalium, sulfur, sugar, and carbonaceous materials.

8.2.7.2 Reaction products of these mixtures are dependent on the original mixture composition and include compounds such as potassium sulfate, potassium chloride, potassium carbonate, potassium sulfide, potassium thiocyanate, potassium bicarbonate, potassium nitrite, potassium chlorate, aluminum oxide, magnesium hydroxide, barium sulfate, and strontium carbonate (**19**).

8.3 *Smokeless Powders:*

8.3.1 If partially-burned particles are present refer to Practice E3253, Practice E2998 and Test Method E2999.

8.3.2 If no particles are present, extract smokeless powder components using an organic solvent and identify by GC-MS. Refer to Practice E2998 and Test Method E2999.

8.4 *Organic High Explosives:*

8.4.1 Organic high explosives can include nitroglycerin (NG), ethylene glycol dinitrate (EGDN), trinitroluene (TNT), cyclotrimethylamine trinitramine (RDX), pentaerythritol tetranitrate (PETN), and cyclotetramethylamine tetranitramine (HMX).

8.4.2 Organic high explosives can be identified within organic solvent extracts, aqueous extracts, or vapor extracts (**20-26**).

8.4.3 Identify organic high explosive residues within extracts by GC-MS or LC-MS. If sufficient residue remains, additional analyses can be conducted. Refer to Practice E3253.

8.4.4 Identification of other components can provide information about the original explosive.

8.5 *Dynamite:*

8.5.1 Dynamite can contain the organic compounds NG and EGDN, as well as inorganic compounds, such as ammonium nitrate and sodium nitrate. Nitrocellulose and fillers, such as wood pulp, can also be present.

8.5.1.1 Military dynamite primarily contains TNT and RDX.

8.5.2 Identify EGDN, NG, or a combination thereof, within an organic solvent extract by GC-MS or LC-MS. If sufficient residue remains, additional analyses can be conducted. Refer to Practice E3253.

NOTE 3—Refer to 8.4 for identifying residues of military dynamite.

8.5.2.1 If proper packaging allows, perform vapor extraction prior to solvent extraction.

8.5.3 Identify residues of inorganic compounds within aqueous extracts by IC-MS or orthogonal IC or CE methods. If sufficient residue remains, additional analyses can be conducted. Refer to Practice E3253.

8.6 *Blasting Agents, Slurries, Water Gels and Emulsions:*

8.6.1 Explosives in this category include some binary explosives, water gels, and emulsions containing ammonium nitrate, sodium nitrate, calcium nitrate, potassium nitrate, or a combination thereof. These materials contain a variety of other components such as water, nitromethane, aluminum, oils, waxes, emulsifiers, gelling agents, or microspheres (glass or phenolic). Sensitizers can be present including some organic high explosives, such as monomethylamine nitrate, or ethanolamine nitrate (**27**).

8.6.2 Refer to 8.4 for identifying residues of organic high explosives that are used as sensitizers.

8.6.3 Identify residues of inorganic salts, monomethylamine nitrate, or ethanolamine nitrate within aqueous extracts by IC-MS, orthogonal IC or CE methods, or derivatization and analysis by LC (**28**). If sufficient residue remains, additional analyses can be conducted. Refer to Practice E3253.

8.7 *Primary Explosives:*

8.7.1 Primary explosives are typically used as initiating explosives in low quantities; therefore, it can be impractical to recover residue. However, some of these primary explosives have been utilized as main explosive charges (**29-36**). These sensitive explosives can include styphnates, azides, fulminates, organic diazo compounds, and organic peroxides.

8.7.2 Refer to 8.4 for identifying residues of primary organic high explosives that do not contain a heavy metal.

9. Documentation

9.1 Retain all notes and supporting analytical data used for an identification in accordance with Practices E860 and E3255. Examples of such data include chromatograms/spectra, photographs/photocopies of results, and detailed descriptions of morphological characteristics.

9.1.1 Case notes should be sufficient to allow an independent analyst to understand and evaluate all the work performed, independently analyze and interpret the data, and form opinions.

9.2 Report results in accordance with E620.

10. Keywords

10.1 explosive residues; explosives; explosives analysis; forensic science; post-blast explosives