Standard Practice for the Collection, Preservation, and Analysis of Organic Gunshot Residues

Gunshot Residue Subcommittee
Chemistry/Instrumental Analysis Scientific Area Committee
Organization of Scientific Area Committees (OSAC) for Forensic Science

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Prepared by
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Subcommittee
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1. Scope

1.1. This standard practice describes procedures for the sampling and preservation of organic gunshot residues (OGSR) recovered from hands, skin, clothing, and other substrates, also instrument conditions for subsequent analysis of samples by gas or liquid chromatography-mass spectrometry (GC-MS or LC-MS), and recommendations for reporting the significance of those findings.

1.2. The analysis of intact smokeless powder grains is beyond the scope of this standard practice.

1.3. OGSR originates from the combustion of the smokeless powder and the priming mixture following their ignition during the firearm discharge process. After a firearm has been discharged the combined residue can be found on exposed surfaces in the vicinity of the fired weapon (i.e. hands, other exposed skin surfaces, hair, clothing, and other surfaces, etc.). OGSR can also be found in the cartridge case after firing and can be recovered to provide information about the constituents of the propellant and/or priming mixture.

1.4. This standard practice discusses the collection, preservation, analysis and interpretation of OGSR. This standard practice does not deal with the analysis of inorganic gunshot residue (IGSR) or primer gunshot residue (pGSR). Individual laboratory protocol will determine if IGSR/pGSR and OGSR will be analyzed in a single technique or separate techniques. The method chosen will dictate sample collection procedures used.

1.5. Additional sources of information on forensic OGSR analysis, not specifically mentioned in the document, may be considered, added or substituted. A review of new sources of information on general forensic methods and forensic OGSR analysis should be carried out on a regular basis (e.g. at least annually) to incorporate well-established current findings and methods into the examination practices and to replace outdated methods.

1.6. This standard practice focuses on the collection, preservation and the analysis of OGSR components using electron ionization (EI)-GC-MS or LC-MS. However, additional analyses may be performed on OGSR components that have been described elsewhere [1,2].

1.7. This standard practice offers a set of instructions for performing one or more specific operations. This standard practice cannot replace knowledge, skill, or ability acquired through appropriate education, training, or experience, and should be used in conjunction with sound professional judgement.

1.8. Units - The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

1.9. This standard practice 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 and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced Documents

2.1. ASTM Standards:

2.1.1 E620 Standard practice for reporting opinions of scientific or technical experts.

2.1.2 E1492 Standard practice for receiving, documenting, storing, and retrieving evidence in a forensic science laboratory.

2.1.3 E1588 Standard practice for gunshot residue analysis by scanning electron microscopy/energy dispersive X-ray spectrometry.

2.1.4 E2329 Standard practice for identification of seized drugs.

2.1.5 E2764 Standard practice for uncertainty assessment in the context of seized-drug analysis.

2.1.6 E2998 Standard practice for characterization and classification of smokeless powders.

2.1.7 E2999 Standard test method for analysis of organic compounds in smokeless powder by gas chromatography-mass spectrometry and Fourier transform infrared spectroscopy.

3. Terminology

3.1 Definitions of terms specific to this standard that are accepted based on consensus of the forensic community:

3.1.1 *Category I, n* – compounds used in the manufacture of smokeless powder or priming compound, and their related degradation products, which are uncommon in other commercial or environmental sources.

3.1.2 *Category II, n* – compounds used in the manufacture of smokeless powder or priming compounds, and their related degradation products, which are common in other commercial or environmental sources.

3.1.3 *Characteristic residues, n.* – Organic residues that have a combination of compounds rarely found in residues from anything other than smokeless powders.

3.1.4 *Consistent residues, n.* – Organic residues that have a combination of compounds that can be found in residues from smokeless powders but also may have other sources.

3.1.5 *Double-base, n* – Smokeless powder containing nitrocellulose and nitroglycerin as the major energetic materials.

3.1.6 *Flash Suppressor, n* – a chemical which, when combusted as part of a propellant mixture, produces gaseous products that dilutes muzzle gases and the associated flash. Flash suppressors are also referred to as flash inhibitors.

3.1.7 *Inorganic GSR (IGSR), n* – Residues from the primer, cartridge case, projectile (e.g. bullet or shot pellets), and/or the firearm that are primarily comprised of metal, metal oxides or metal salts.

3.1.8 *Primer GSR (pGSR), n* –Residues generating from the priming mixture that could be inorganic or organic in nature.

3.1.9 *Plasticizer, n* – Compound used to promote plasticity of a material, including flexibility, compressibility and reduce brittleness.

3.1.10 *Organic GSR (OGSR), n* – Residues from the propellant and the priming mixture that are organic (carbon-based) in nature.

3.1.11 *Oligomer, n* – A polymer whose molecule consist of relatively few repeating units.

3.1.12 *SEM-EDS, n* – Scanning Electron Microscope with Energy Dispersive X-ray Spectroscopy.

3.1.13 *Sensitizer, n* – a chemical material added with the purpose of aiding in the initiation or propagation of a detonation. This additive will lower the ignition temperature of the propellant mixture, or if added to the primer, will increase the impact sensitivity of the primer composition (i.e. lower energy threshold required to ignite by impact).

3.1.14 *Single-base, n* – Smokeless powder containing nitrocellulose as the major energetic material.

3.1.15 *Smokeless powder, n* – A propellant and low explosive composed of nitrocellulose and other organic and inorganic compounds.

3.1.16 *Stabilizer, n* – A compound or preservative used to prevent or slow down decomposition.

3.1.17 *Stubbing, v* – Act of pressing adhesive tape onto surface to be sampled using a collection stub; synonymous with dabbing or tape lift.

3.1.18 *Triple-base, n* – Smokeless powder containing nitrocellulose, nitroglycerin and nitroguanidine.

4. Significance and Use

4.1. Gunshot residue (GSR) examination is generally conducted to determine if an individual has discharged a firearm, was in close proximity to a discharged firearm, or came into contact with an object having gunshot residue on its surface. Traditional GSR analysis has relied upon the detection of inorganic GSR primarily originating from the ammunition primer (pGSR); however, OGSR provides information that complements pGSR analysis [1]. This standard practice is of use to forensic laboratories desiring to supplement pGSR analysis with OGSR analysis to identify surfaces exposed to gunshot residue.

4.2. This document provides a standard practice for OGSR analysis using GC-MS or LC-MS. Simultaneous analysis of OGSR and pGSR is outside the scope of this document. However, there are several papers that present a viable method for simultaneous processing and analysis [3,4]. Therefore, the laboratory must decide how to proceed if analyzing OGSR and/or pGSR separately, when only one combined sample has been received (see section 8.3.1.2 for recommendations provided in this practice).

5. Summary of Practice

5.1. This standard practice describes techniques for collecting OGSR samples from different substrates, appropriate sample preservation techniques, and analytical procedures used to analyze OGSR. The interpretation and significance of these results is also addressed.

6. Apparatus

6.1. Gas chromatography-mass spectrometry (GC-MS) – a gas chromatograph (GC) that uses capillary columns and can be interfaced to a mass spectrometer (MS) operating in electron ionization (EI) mode.

6.2. Liquid chromatography-mass spectrometry (LC-MS) – a liquid chromatograph (LC) using a reversed phase column and pump system providing a gradient of at least two solvents (small column diameters, low flow rates for high sensitivity) coupled to a mass spectrometer with electrospray ionization (ESI) or atmospheric pressure chemical ionization (APCI) ion source working in positive and negative ion mode. Mass analyzers with higher resolution and mass accuracy, with mass measurements to the 4th decimal place, are recommended (e.g. time-of-flight, triple quadrupole, etc.)

7. Materials

7.1. Solvents –LC-MS grade or a higher grade solvents (acetone, acetonitrile, ethanol, isopropanol, methanol, water, or other suitable solvents) should be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society [5]. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity without any contaminants to permit its use without lessening the accuracy of the determination.

7.2. Sample Collection Solvents – acetone, acetonitrile, ethanol, isopropanol or methanol or other appropriate solvents of LC-MS grade or equal quality are acceptable.

7.3. OGSR Standard(s) or Reference Materials – Certified reference materials should be purchased in a suitable grade, whenever possible. If an OGSR component is not available as a certified standard, ACS grade or higher should be purchased.

7.3.1 Appropriate concentration of individual reference materials or standards of these compounds may be used in addition to or instead of a reference standard.

7.4. Internal Standard – Not recommended for qualitative identification of OGSR.

7.5. GC Carrier Gas - Helium or hydrogen of purity 99.995% or higher.

8. Procedure

8.1. Collection of OGSR Samples – There are three commonly accepted sampling techniques for OGSR. However, each technique differs in its collection efficacy [6]. Whichever technique is applied, a separate sampling device should be used for each surface (e.g. left hand, right hand, etc.) to maximize sample recovery. [6,7]

8.1.1 Swabbing

8.1.1.1 Dry or wet swabs can be used to sample skin (e.g. hands, face, neck), surfaces, or objects. If wet swabbing, suitable solvents include ethanol, acetone, isopropanol or acetonitrile (not recommended for skin). Suitable swabs include cotton, polyester, muslin or nylon-based substrates [6,8]. A note of caution in choosing the solvent, the solvent used may dissolve other compounds that could interfere with the analysis. [6,7,8]

8.1.1.2 If a surface is to be stubbed for recovery of pGSR, the adhesive tape lift should be used first and then swabbed with a suitable swab as defined above. [6]

8.1.2 Adhesive tape lifts

8.1.2.1 Adhesive tape lifts used for SEM sample stubs effectively collect OGSR from many surfaces, including skin, clothing and objects. Other adhesive tapes can be used at the discretion of the individual laboratory. [4,9]

8.1.2.2 Dab the adhesive surface of the stub onto the surface to be sampled until tackiness is lost to achieve maximum collection efficiency. [10]

8.1.3 Vacuuming

8.1.3.1 Vacuum lifting is appropriate for clothing or porous objects. A suitable device requires a vacuum fitted with an inert filter membrane with a maximum porosity of 0.5 µm to trap particulates. Teflon or fiberglass filters are suitable. [7]

8.2 Preservation of OGSR Samples

8.2.1 Samples (swabs, adhesive lifts and vacuum filters) should be stored in airtight containers immediately to reduce possible loss due to evaporation. Samples should be stored at 0°C or less to maximize preservation.[6,8]

8.3 Extraction of OGSR

8.3.1 Solvent Extractions

8.3.1.1 Swabs, filters, stubs or other adhesive lifts that have not been coated or analyzed by SEM-EDS, can be extracted using a suitable organic solvent, such as methanol, methylene chloride or acetonitrile. Extracts should be sonicated at least 5 minutes and then concentrated by gently evaporating with nitrogen gas to near dryness, prior to analysis. Extracts may be reconstituted to an appropriate volume using an organic solvent that is of suitable purity for GC-MS or LC-MS analysis.[7]

8.3.1.2 Adhesive stubs that have already been prepared and analyzed by SEM-EDS for pGSR, can be subsequently extracted to recover OGSR. [10]

8.3.1.3 Extraction from stubs and other adhesive lifts should be performed using either an 20/80 ethanol: water mixture or methanol to mitigate interferents from the adhesive. [11]

8.4 GC-MS Analysis of OGSR Extracts

8.4.1 GC-MS analysis will identify the common organic components found in OGSR, with the exception of nitroguanidine and nitrocellulose, unless derivatized. [12,13,14]

8.4.2 An example of GC-MS parameters suitable for OGSR analysis is provided in Table A1.

8.4.3 Column length and temperature program conditions may be modified to specific user requirements, provided that each peak for a given compound of a test mixture, reference material, or appropriate standard is resolved.

8.4.4 Table 1 describes the target compounds that could be used to identify the presence of OGSR components and their key ions. For identification requirements please refer to section 10.1.

TABLE 1: Target Compounds for GC-MS analysis and m/z of key ions in EI MS for smokeless powder residues

Target Compound	**Significance/ Application**	**Target Ions (m/z)**
Akardite II (AK II)	Stabilizer	169, 226
2-amino-4,6-dinitrotoluene (2-ADNT)	Degradation products	197, 180
Dibutyl phthalate (DBP)	Plasticizer	149, 223
Diisoamyl phthalate (DIP)	Plasticizer	149
2,4- and 2,6-dinitrotoluene (2,4-DNT and 2,6-DNT)	Flash suppressor	165, 182
Dioctyl phthalate (DOP)	Plasticizer	149, 279
Diphenylamine (DPA)	Stabilizer	169
Ethyl centralite (EC)	Stabilizer	120, 268
Ethyleneglycol dinitrate (EGDN)	Explosive	46, 76
Methyl centralite (MC)	Stabilizer	134, 240
2 and 4-nitrodiphenylamine (2-NDPA, 4-NDPA)	Degradation products; stabilizer	167, 214
Nitroglycerin (NG)	Explosive	46, 76
Nitroguanidine (NQ)	Explosive	58, 104
4-nitrosodiphenylamine (4-NODPA)	Degradation products	167, 198
N-nitrosodiphenlyamine (N-NODPA)	Degradation product	Detected as DPA
Nitrotoluenes (NTs)	Sensitizer	65, 91, 120 or 137
2,4,6- trinitrotoluene (TNT)	Flash suppressor/sensitizer	89, 210

8.5 LC-MS Analysis of OGSR Extracts

8.5.1 LC-MS analysis will identify the common organic components found in OGSR. Nitrocellulose is only detected in rare cases as an oligomer.

8.5.2 Only samples of appropriate dilution should be analyzed on a LC-MS system. Filtration or centrifugation of extracts and/or the use of a guard column is recommended.

8.5.3 Table A2 lists example LC-MS parameters that can be used to analyze OGSR.

8.5.4 Different columns, flow rates, and solvent gradients require individual parameter optimization using a standard mixture for a specific LC-MS setup.

8.5.5 Table 2 describes the common target compounds that could be used to identify the presence of OGSR components and their key ions.

TABLE 2: Target Compounds for LC-MS analysis and m/z of key ions in MS for smokeless powder residues

Target Compound	**Mode**	**Precursor Mass (*m/z*)**	**Ionization**	**Ref.**
Nitroglycerin (NG)	ESI neg., APCI neg.	227.0026 + adduct mass	[M+adduct]- adduct to be determined by user	[15]
Nitrocellulose (NC); trimethylsilyl derivatized	ESI neg., APCI neg.	481.2294	$[M-H]^-$	[13]
Diphenylamine (DPA)	ESI pos. APCI pos.	170.0971	$[M+H]^+$	[4]
2 and 4-nitrodiphenylamine (2-NDPA, 4-NDPA)	ESI pos. APCI pos.	215.0821	$[M+H]^+$	[4]
N-nitrosodiphenylamine (N-NODPA)	ESI pos. APCI pos	199.0872	$[M+H]^+$	[16]
2,4- and 2,6-dinitrotoluene (2,4-DNT and 2,6-DNT)	ESI neg	181.0250	$[M-H]^-$	[4]
Methyl centralite (MC)	ESI pos, APCI pos.	241.1342	$[M+H]^+$	[4]
Ethyl centralite (EC)	ESI pos, APCI pos.	269.1655	$[M+H]^+$	[4]
Akardite II (AK II)	ESI pos, APCI pos.	256.1087	$[M+H]^+$	[16]

9. Quality Control

9.1 Minimum quality assurance protocols

9.1.1 Establish protocol to confirm optimum instrument calibration and parameters on a routine basis.

9.1.2 A method blank should be prepared using the same procedure(s), reagents and conditions for analysis with questioned extracts.

9.1.3 A reference mixture of selected components listed in Tables 1 and 2, as chosen by the individual laboratory for the associated instrumentation, shall be analyzed with questioned extracts. The reference mixture must be stored in a suitable container under appropriate conditions. It is recommended to establish a protocol to ensure stability of the reference mixture and that it be replaced if degradation is suspected.

10. Data Analysis

10.1 Identification

10.1.1 The following minimum criteria must be used when making chemical compound identification(s): [17,18]

10.1.1.1 The analytical scheme must include at least two techniques; gas chromatography-mass spectrometry or liquid chromatography-mass spectrometry.

10.1.1.2 Laboratories shall establish acceptance criteria for each technique used based upon validation data, and include measurement uncertainty limits if quantitative analysis is being performed.

10.1.1.3 The use of two techniques (e.g. GC-MS) requires that the individual acceptance criteria for each technique are fulfilled, separately, and the results from both are used in making the identification; e.g. retention time for GC and LC along with fragment masses for MS.

10.1.1.4 Data from a test result shall be compared to data from a reference material run under the same instrumental conditions.

10.2 Classification

10.2.1 The significance of compounds identified (Table 2) is based on their observed association with OGSR and with other environmental sources unrelated to OGSR. A classification scheme (Table 3) is proposed based on a review of relevant literature. [19,20,21]

TABLE 3: Classification of compounds associated with OGSR

Category	**Compounds**	**Function**
1	EC	Stabilizer
	MC	Stabilizer
	NG	Explosive
	Nitroguanidine (NQ)	Explosive
	2-, 4-NDPA	Stabilizer
	N-NODPA	Degradation Product
2	NC	Explosive
	Other nitrotoluenes	Sensitizer
	DPA	Stabilizer
	Other DPA derivatives	Stabilizer
	2,4-DNT	Flash Suppressor
	Ak II	Stabilizer

10.3 Analytical Conclusions

10.3.1 Based on the classification scheme for compounds identified (Table 3), minimum criteria to report OGSR has been identified are:

10.3.1.1 At least two category I compounds, with or without a category II compound, the residue is characteristic of OGSR;

10.3.1.2 One category I compound and at least one category II compound, the residue is consistent with OGSR.

10.3.2 If less than the minimum is identified, each compound identified may be listed but an inconclusive result should be reported. The reason for the inconclusive results shall be included for any inconclusive result.

10.3.3 If no OGSR components are detected, a negative result should be reported.

10.3.4 Additional reporting criteria with associated analytical conclusions can be developed for combinations not included in the previous sections. Any new criteria should aid in differentiating environmentally or occupationally produced residues that could be found in an OGSR sample. An assessment of the significance of these combinations and conclusions shall be made in consideration of appropriate research and documentation.

10.4 Limitations

10.4.1 Certain OGSR components can persist with time, depending on the volatility of the compound and adhesion or absorption by the particular substrate. [8]

10.4.2 The common mechanisms for loss of OGSR are permeation (into the skin), evaporation (from the skin) or physical removal (e.g. wiping, skin shedding). The extent of loss will vary, but generally, OGSR will persist for longer periods on porous or absorptive surfaces. [22]

10.4.3 Certain OGSR components can be recovered from different materials unrelated to firearms, such as additives (plasticizers, stabilizer) or explosives (NG and NC), and can also be transferred between surfaces by physical contact (i.e. secondary-transfer). [6] A statement to acknowledge these limitations should be included with the report or supplementary information appended to a report.

10.4.4 OGSR identified on one or both hands of a person sampled, cannot be used to infer if one or the other hand held a firearm or was used to fire a firearm.

11. Documentation

11.1 Hardcopies and/or electronic format for the following must be generated and should be retained for an appropriate period of time as determined by the individual laboratory:

11.1.1 Instrument parameters that were used for analysis for the applicable instrument (GC-MS or LC-MS).

11.1.2 Documentation of calibration and tuning of the applicable instrument used for analysis (GC-MS or LC-MS).

11.1.3 Chromatograms, to include retention times of blank(s), reference material(s) and questioned samples.

11.1.4 Mass spectra of OGSR compounds identified and the associated reference material(s) used for comparison to identify.

11.1.5 All analytical notes from the examiner regarding OGSR analysis.

12. Keywords

12.1 OGSR; GC-MS; LC-MS

13. References

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ANNEX

(Mandatory Information)

A1. INSTRUMENTAL OPERATION PARAMETERS

A1.1 GC-MS parameters example

A1.1.1 An example GC-MS instrumental method, from literature, to analyze OGSR compounds is provided below as a starting point for the laboratory validation process.

TABLE A1: Recommended GC-MS conditions [14]

Parameter	**Condition**
GC column:	30 meter, DB-5
Injector Temp:	180° C
Initial Temp (T_0):	40-50° C
Hold time (T_0):	3 minutes
Ramp 1:	15° C/minute to 150° C (T_1)
Hold time (T_1):	2 minutes
Ramp 2:	20-40° C/minute to 265° C (T_2)
Hold time (T_2):	8 minutes
Mode:	EI
Scan Mode	Full Scan or Selected Ion Monitoring
Split Ratio:	Splitless (n/a)
MS range:	40 to 500 *m/z*
Source Temp:	230° C
Solvent delay:	3 minutes

A1.2 Recommended LC-MS Parameters

A1.2.1 An example LC-MS instrumental method, from literature, to analyzed OGSR compounds is provided below as a starting point for the laboratory validation process. This method uses a quadrupole-time of flight mass spectrometry (qTOFMS); however, this method can be modified for any mass spectrometer system.

TABLE A2: Recommended LC-MS conditions [4]

Parameter	**Condition (APCI)**	**Condition (ESI)**
LC column	C18 column: 100 mm × 3 mm, 2.6 µm C8 column: 2.1 × 150 mm, 5 µm	C18 column: 100 mm × 3 mm, 2.6 µm C8 column: 2.1 × 150 mm, 5 µm

Column Temperature	40 ± 5 °C	40 ± 5 °C
Elution Gradient Solvent A Solvent B	 Ammonium acetate 1 mM Methanol	 Ammonium acetate 1 mM Methanol
Injection Amount	5 µL	5 µL
Needle Rinse	90 s with methanol	90 s with methanol
MS Ionization Interface	APCI	ESI
Ionization Gas	Dichloromethane	Dichloromethane
Dry Gas Flow	15 L/min	10 L/min
Dry Gas Temperature	300 °C	300 °C
Nebulizer Pressure	30 psi	30 psi
Sheath Gas Temperature	300 °C	300 °C
Ionization Mode	Positive and Negative applied alternatively, Positive, or Negative	Positive and Negative applied alternatively, Positive, or Negative