TWGFEX Laboratory Explosion Group
Standards & Protocols Committee

Recommended Guidelines for Forensic Identification of Post-Blast Explosive Residues

1.1 Introduction:

The purpose of this document is to set forth guidelines for the forensic identification of post-blast explosive residues. It is recognized that the correct characterization and/or identification of an explosive residue depends on the use of scientifically acceptable analytical methods and the expertise of the analyst. Unique requirements in different jurisdictions may dictate practices followed by a particular laboratory. This document does not discourage the use of any particular method within an analytical scheme and recommends the use of multiple techniques based on different principles and methodologies. Analytical chemistry is an advancing science and there are other analytical techniques which are not listed in the document but which may be employed in the identification of post-blast explosive residues.

1.2 Categorizing Analytical Techniques:

For purposes of this document, techniques for the analysis of post-blast explosive residues may be broken down into four categories: (1) those that provide significant structural and/or elemental information, (2) those that provide limited structural or elemental information, (3) those that provide a high degree of selectivity, and (4) those that are useful but do not fall in either of the other categories. Table 1 lists examples of such techniques based on the 1999 TWGFEX survey of explosion debris analysts in the United States.

Table 1: Categories of Analytical Techniques

Categories 1 and 2	Category 3	Category 4
Infrared Spectroscopy (IR)	Gas Chromatography (GC)	Flame Test
Gas Chromatography/Mass Spectrometry (GC/MS)	Gas Chromatography Thermal Energy Analyzer (GC-TEA or EGIS)	Spot Test
Energy Dispersive X-Ray Analyzer (EDX)	Liquid Chromatography (LC)	Melting Point
Raman Spectroscopy	Liquid Chromatography Thermal Energy Analyzer (LC-TEA)	
X-Ray Diffraction (XRD)	Ion Chromatography (IC)	
Liquid Chromatography/ Mass Spectrometry (LC/MS)	Capillary Electrophoresis (CE)	
	Thin Layer Chromatography (TLC)	
	Ion Mobility Spectrometry (IMS)	
	Polarizing Light Microscopy (PLM)	
	Stereo Light Microscopy (SLM)	

1.3 Recommended Practices:

1.3.1 Good laboratory practices suggest that multiple techniques be employed in forensic post-blast explosive residue identification and that supporting analytical data be reviewable. Examples of such data include printed chromatograms, photographs/photocopies of results, or detailed descriptions of morphological characteristics. For any given component, the identification should be no less rigorous than the criteria found in The TWGFEX Guideline for the Forensic Identification of Intact Explosives (July 2004).

1.3.2 If a method exists in the laboratory for the collection and analysis of volatile explosives and the exhibits have been properly packaged since collection at the scene (i.e. clean, properly sealed, airtight containers), conduct volatile sampling prior to opening the containers for other examinations.

1.3.3 If applicable, conduct a macroscopical examination of the debris from the seat of the explosion and any visible residue in the vicinity. A microscopical examination of the area of interest is then conducted to determine if particulate matter is present which could be analyzed directly. The degree of usefulness of these initial observations assumes the examiner has experience in recognition of post-blast explosive debris, residues and the associated physical damage. While the visual examination may be suggestive of the use of an explosive, it is necessary to use additional analytical techniques to identify the explosive compound itself, its key constituents or its residues. If particles of uninitiated explosive are found, follow the TWGFEX Guideline for the Forensic Identification of Intact Explosives (July 2004). If particulate matter if available, utilize suitable analytical methods to identify the components. An attempt should be made to identify the particulate matter prior to moving on to extractions. Suitable analytical techniques include FTIR, microcrystalline tests, PLM, IMS, EGIS, EDX, Raman, melting point and flame tests, although EDX and flame tests are not usually useful for characterizing organic materials.

1.3.4 In the absence of particulate matter or if the analyses of the particulate matter did not identify the material, extractions of the debris should be made. Normally, aqueous extracts would be analyzed by IC, CE, LC, spot tests, microcrystalline tests and flame tests. Evaporated aqueous extracts are suitable for FTIR, XRD, EDX, Raman and PLM. Solvent extracts are normally analyzed by LC/MS, GC/MS, GC-TEA, EGIS, IMS, GC-ECD, FTIR and TLC. Suitable organic solvents in use are acetonitrile, acetone, ethanol, methanol, methylene chloride and ethyl acetate.

1.3.5 For an analytical technique to be considered of value, the test must be considered ‘positive’. While ‘negative’ tests provide useful information for ruling out the presence of a particular family of explosives, negative results have limited value toward establishing the identification of an explosive substance. (i.e. a white crystal tested negative for nitrates with spot tests; this steered the investigation toward improvised explosives even though it failed to identify the material)

1.4 Interpretation of Results:

1.4.1 Appendix A is a chart showing a list of explosives and their residues. It is possible to find uninitiated explosive as well as reaction products in a post-blast situation. (The chart is not necessarily all-inclusive but the species mentioned are known to have been identified in post-blast debris).

1.4.2 Compiling a list of the materials identified is recommended, especially in the absence of unreacted materials. A review of the Appendix chart of explosives and their residues will help the analyst decide which explosives are possible sources. Since many post-blast materials are common to more than one explosive, it may be necessary to include more than a single source in the conclusion.

1.5 Post-Blast Training Exercises:

Forensic laboratories are encouraged to participate in controlled explosions of known explosives and the subsequent analysis of the post-blast debris to determine if their analytical methods are capable of detecting and identifying the explosives or their residues in post-blast situations.

1.6 Safety:

The laboratory is strongly encouraged to establish protocols for the safe execution of these procedures.

1.6.1 Summary:

It is understood that the forensic identification of post-blast explosive residues may be accomplished by a variety of analytical techniques.

These guidelines were formulated with the express intent of application to post-blast residues from both commercial and improvised explosives.

APPENDIX A
Explosives and Their Post-Blast Residues

EXPLOSIVE	UNINITIATED	OBSERVATION	POST-BLAST/BURN
Black Powder	KNO_3, C, S	Sulfur smell, white smoke, visible grey residue	SO_4^{2-}, NO_2^-, CO_3^{2-}, SCN^-, OCN^-, S^{2-}, K^+, $S_2O_3^{2-}$
Pyrodex	KNO_3, C, S, DCDA, SB, $KClO_4$	Less sulfur smell and residue than black powder	SO_4^{2-}, CO_3^{2-}, Cl^-, benzoate, DCDA, OCN^-, NO_2^-, K^+,
Triple Seven	KNO_3, C, DCDA, SB, $KClO_4$, 3-NBA	No sulfur smell	CO_3^{2-}, Cl^-, K^+, NO_2^-, benzoate, DCDA, OCN^-
Black Canyon/Clean Shot Pioneer/Golden Powder	KNO_3, C, S, ascorbic acid	Sulfur smell, white smoke, visible grey residue	SO_4^{2-}, **NO_2^-**, **CO_3^{2-}**, **S,K** SCN^-, OCN^-, $S_2O_3^{2-}$
Flash Powder	Fuel [Al, Mg, S] and oxidizer	Silvery residue	ClO_4^-, ClO_3^-, metals, oxides
Smokeless (single base)	NC		Unreacted particles, NC
Smokeless (double base)	NG, NC		Unreacted particles, NC, NG
Dynamite	NG ester, AN/SN, other salts		Unreacted material, NG, EGDN, AN/SN (or ions)
PETN, RDX, HMX, EGDN, HMTD, Tetryl	n/a		Parent compound found
TNT	TNT and often DNT's		TNT and often DNT's

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Water gels and Slurries	Inorganic nitrate and sensitizer		NO_3^-, MMA $^+$,
Emulsions	Inorganic nitrates, waxes, sensitizers	**Al, smokeless powder particles & perchlorates, are sometimes present**	NO_3^-,
ANFO	AN and fuel oil		NO_3^-, NH_4^+, fuel oil, NO_2^-
Binaries	AN, nitromethane	Some have visible dyes	NH_4^-, NO_3^-
PBX	Explosive, binder, plasticizer	Black soot, some have dyes	Unreacted materials
Primary explosives	Metal + N_3, **(ONC)**$_2$, $C_6H_3O_8N_3$	Typically involve heavy metals such as lead, mercury, silver	Unlikely to survive (maybe lead from lead azide, etc)
Improvised Explosives/Incendiaries			
TATP	$C_9H_{18}O_6$	Peracetic acid, acetone, uninitiated material	Peracetic acid, acetone
HMTD	$C_6H_{12}N_2O_6$	Uninitiated material	Hexamine
Dry ice + water in bottle	CO_2	delay - no smoke	No chemical evidence, characteristic broken edges of container
Acetylene/natural gas/propane (often in a plastic bag)	C_2H_2	lots of soot, lot of destruction,	Container fragments, fuse remains, odor?

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Pool chlorine & an alcohol	$Ca(OCl)_2$, $CaCl_2$, $Ca(OH)_2 \cong 2H_2O$	delay mechanism that can blow up if confined	Cl_2 odor
Pool chlorine and brake fluid	$Ca(OCl)_2$, $CaCl_2$, $Ca(OH)_2 \cong 2H_2O$	delay mechanism that can blow up if confined, white smoke, fireball	Cl_2 odor
Pool chlorine and glycerin	$Ca(OCl)_2$, $CaCl_2$, $Ca(OH)_2 \cong 2H_2O$	delay mechanism that can blow up if confined, fireball, white smoke	Cl_2 odor, $CaCl_2 \cong 6H_2O$ and $CaCO_3$
Pool chlorine & milk products	$Ca(OCl)_2$, $CaCl_2$, $Ca(OH)_2 \cong 2H_2O$	delay mechanism that can blow up if confined, white smoke, sour milk smell	Cl_2 odor,
Pool chlorine and oil (drying oils, vegetable oils, etc)	$Ca(OCl)_2$, $CaCl_2$, $Ca(OH)_2 \cong 2H_2O$	delay mechanism that can blow up if confined	Cl_2 odor, $CaCl_2 \cong 6H_2O$, $CaCO_3$, $CaCl_2$ $Ca(OCl)_2$, $Ca(OH)_2 \cong 2H_2O$, butyric acid?
Pool chlorine + antifreeze	$Ca(OCl)_2$, $CaCl_2$, $Ca(OH)_2 \cong 2H_2O$	delay mechanism that can blow up if confined, fireball, white smoke	Cl_2 odor,
Pool chlorine (organic) and alcohol	R-OH only works with organic pool product	delay mechanism that can blow up if confined, grayish white smoke	Cl_2 odor,
ANTI (NI_3)	n/a		Find NH_4I and yellow stain (I_2), spattering of unreacted material

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Chlorate & sugar	n/a		KCl or NaCl, residual sugar, odor of burnt sugar, ClO_3^-
Paper match heads (safety matches)	$KClO_3$, S, SiO_2	sulphur smell	KCl, SiO_2, paper fibers, unreacted match heads, S, $SO_4 2^-$, S_2^-,
Strike anywhere match heads	P, S, $KClO_3$, SiO_2	sulphur smell	Unreacted match heads, P, S, $SO_4 2^-$ S_2^-,
Red phosphorus and chlorate (Armstrong's mixture)	P, $KClO_3$		P, ClO_3^-, K, Cl^-, PO_4^{3-}
HCl and aluminum	HCl, Al, H_2O or Etchant ($FeCl_3$ + H_2O) and Al foil	'acid' odor	$Al_2Cl_6.6H_2O$ and H_20, unconsumed Al, acidic
HotPack or ActionPack	Magnesium iron alloy, NaCl, silica and wetting agent	steam	Magnesium hydroxide
Tannerite (see website for use)	Al, finely divided AN	Silvery residue	Unreacted materials, NO_2^-, NO_3^-, NH_4^+, Al
Mischmetal	Ce, La, Nd, Fe	sparks on impact	Sparks and unreacted material
Urea nitrate	n/a		Unreacted material, NO_2^-, NO_3^-

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Al and I_2	n/a	purple smoke,	Unreacted materials, brown/purple residue
50:50 Zn:S (both powders)	Zn , S	burns fast/explode in container, grey smoke	Zn, S, ZN^{+}, $S^{=}$
CCl_4 + Al powder (1:4)	CCl_4 , Al	detonates	Al
Vaseline and $KClO_4$ (1:2)(poor man's C-4)	$KCLO_4$, vaseline	dark grey smoke needs detonator	$ClO4^{-}$, unreacted material
$KMnO_4$ + glycerin	$KMnO_4$, glycerin	delay, deep purple smoke	Unreacted materials
Drano + aluminum	NaOH , Al	delay, extra Al makes it work faster, generates H_2	Al, Na
Al + lye/drano + water	Al , KOH , NaOH , H_2O	generates H_2	Al(OH), NaOH, basic
Al + HCl	Al , HCL	generates H_2	Al, Al^{+}, Cl^{-}, acidic

Abbreviations Used :

ECD	electron capture detector
FID	flame ionization detector
PDA	photo-diode array (detector)
TEA	chemiluminescence detector
EGIS	fast GC with chemiluminescence detector
3-NBA	3 nitro-benzoic acid
SB	sodium benzoate
DCDA	dicyanodiamide
PBX	plastic bonded explosives
SN	sodium nitrate
AN	ammonium nitrate
MMA	monomethylamine
MMAN	monomethylammonium nitrate
EA	ethanolamine
EDDN	ethylenediamine dinitrate