APPENDIX A – SPOT TEST PROCEDURES (ATF)

TABLE OF CONTENTS

SYSTEMATIC ANALYSIS OF LOW EXPLOSIVES

		Page
SUMMARY OF RESULTS:		
	Anions	**1**
	Cations and Miscellaneous	**2**
ANION SPOT TESTS:		
	Carbonates	**3**
	Chlorates	**4**
	Chlorides	**5**
	Nitrates	**6**
	Nitrites	**7**
	Perchlorates	**8**
	Permanganates/Manganous Salts	**9**
	Phosphates	**9**
	Sulfates	**10**
	Sulfides	**10**
CATION AND MISCELLANEOUS SPOT TESTS:		
	Aluminum	**12**
	Ammonium	**12**
	Antimony	**13**
	Barium	**13**
	Carbon	**14**
	Iron	**14**
	Magnesium	**15**
	Phosphorus	**15**
	Potassium	**15**
	Sodium	**16**
	Strontium	**16**
	Sugar	**17**
	Sulfur	**17**
	Zinc	**18**
	CHART SUMMARIZING METAL SOLUBILITIES	**18**

RETIRED

SOURCES OF CHEMICALS: **19**
REFERENCES: **20**

RETIRED

Summary of Results
Systematic Analysis of Low Explosives – Anion Spot Tests

ANION	REAGENT METHOD	MDL – Minimum Detection	Interferences & Comments	POSITIVE CRITERIA
Carbonates	Barium Chloride Acid Uranyl Ferrocyanide	mg 0.0001 mg 0.1 mg	Sulfates* Bicarbonates* Basic solution*	Soluble wt ppt Effervescence Decolorization
Chlorates	Aniline Sulfate Diphenylamine Indigo	mg 0.001 mg 0.0001 mg	* Many oxidiz* Nitrates*	Blue color Blue color Decolorization
Chlorides	Silver Nitrate Oxine/Silver Nitrate	0.01 mg –	Basic, Halides*	White ppt. Turbid., ppt.
Nitrates	Modified Griess/Zn Nitron Diphenylamine	mg mg 0.01 mg	Very basic* ClO_4 Many oxidiz.*	Pink/red Needle crystal Blue color
Nitrites	Modified Greiss Diphenylamine	0.0001 mg 0.001 mg	Very basic* Many Oxidiz.*	Pink/red Blue color
Perchlorates	Triphenylselenium Cl Nitron Methylene Blue	0.1 mg 0.1 mg 1.0 mg	Slow to form* Slow, nitrates*	Crystals Blade crystal Purple color
Permanganates	Filter Paper Ammonium Persulfate/S	– –	Slow	Brown/black Red/violet
Phosphates	Commercial Test Kit Ammonium Molybd/SnCl	0.1 mg 0.001 mg	Sulfides*	Blue color Blue color
Sulfate	Barium Chloride Sodium Rhod/Barium Cl	0.1 mg 0.1 mg	Carbonate*	White ppt. Decolorization
Sulfide	Sodium Azide Lead Acetate Paper Fuchsin	0.1 mg 1.0 mg 0.1 mg	Basic, thios.* Hydrosulfide* Sulfite*	Decolorization, gas Brown-black Decolorization

Notes: Refer to text for description of interferences

All aqueous solutions prepared with distilled or deionized water.

All UV performed at long (366 nm) wavelengths.

MDL = Minimum Detection Level = approximate quantity of the POTASSIUM salts which reproducibly produces positive results.

The technique and experience of the analyst, as well as preconcentration by partial evaporation of solvents, can greatly affect the detection of these anions.

*Tested for reactions and interferences with reagent solutions, including but not limited to the following: Acetic acid, ammonium hydroxide, antimony chloride, barium nitrate, hydrochloric acid, hydrogen peroxide, nitric acid, phosphoric acid, potassium carbonate, potassium chlorate, potassium chloride, potassium nitrate, potassium nitrite, potassium perchlorate, potassium sulfate, sodium sulfide, sucrose, and sulfuric acid.

RETIRED

Summary of Results
Systematic Analysis of Low Explosives – Cation and Miscellaneous Spot Tests

CATION AND MISCELLANEOUS	REAGENT METHOD	MDL – Minimum Detection	Interferences & Comments	POSITIVE CRITERIA
Aluminum	Acid/Base Solubility Morin	– –	Pb, Zn** Tin, Basic**	Sol base, acid UV green fluor.
Ammonium	Nessler Commercial Test Paper	0.01% NH_4OH 0.01%	 *	Orange/brown Brown
Antimony	Rhodamine B		Sulfide*	Violet
Barium	Sodium Rhodizinate	0.1 mg $BaNO_3$	Sr, FRESH RGT!*	Red ppt.
Carbon	Visual/Ignition	–		Black/glowing
Iron	Acid solubility Potassium Thicyanate Dipyridyl	– – –	** PO_4 NO_3*	Sol HCl, yellow Red ppt (FE III) Pink/red (FE II)
Magnesium	Acid Solubility Quinalizarin	– –	**	Sol dilute acid Change to blue
Phosphorus	Oxidize to phosphate	–		See phosphate
Potassium	Commercial Test Paper Zinc Uranyl Acetate	0.01 mg KNO_3 >1.0 mg	Ammonium Sodium	Orange Needle crystals
Sodium	Zinc Uranyl Acetate	0.1 mg $NaNO_3$	Conc. Potassium	UV green fluor.
Strontium	Flame Test Sodium Rhodizonate	 0.1mg $SrNO_3$2	 Ba, FRESH RGT!	Crimson red Red ppt.
Sugar	Napthol	0.1 mg Sugar	(others pink)	Purple
Sulfur	Pyridine/NaOH Benzoin/Lead Acetate	0.001mg S –	Water, contam.	Yellow/blue/brown
Zinc	Acid Solubility	– –	**	Sol. Conc. Acid Red ppt.

Notes: Refer to text for description of interferences.

All aqueous solutions prepared with distilled or deionized water.

All UV performed at long (366 nm) wavelengths.

MDL = Minimum Detection Level = approximate quantity of the LISTED REAGENT which reproducibly produces positive results.

The technique and experience of the analyst, as well as preconcentration by partial evaporation of solvents, can greatly affect the detection of these cations.

*Tested for reactions and interferences with reagent solutions, including but not limited to the following: Acetic acid, ammonium hydroxide, antimony chloride, barium nitrate, hydrochloric acid, hydrogen peroxide, nitric acid, phosphoric acid, potassium carbonate, potassium chlorate, potassium chloride, potassium nitrate, potassium nitrite, potassium perchlorate, potassium sulfate, sodium sulfide, sucrose, and sulfuric acid.

** See chart summarizing metal solubilities on page 18.

RETIRED

SYSTEMATIC ANALYSIS OF LOW EXPLOSIVES
ANION SPOT TESTS

CARBONATES

Barium Chloride

Procedure:
One or two drops of the unknown solution is placed in a black spot plate. One drop of barium chloride solution is added. A white precipitate which is soluble in concentrated acetic acid indicates the presence of carbonate ions.

Reagents:
5g of Barium Chloride in 100 mL of water
Concentrated Acetic Acid

Notes: See **sulfates**. A precipitate of barium sulfate will not dissolve in concentrated acetic acid, while a barium carbonate precipitate will. Barium chloride is **POISONOUS.**

Acid

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. Concentrated HCl is added dropwise. Effervescence indicates the presence of carbonate ions.

Reagents:
Concentrated Hydrochloric Acid

Notes: Bicarbonates react similarly, as do sulfides, sulfites, and thiosulfates; however the latter release gases with distinctive odors. This test does not have to be run independently, simply noting any effervescence with strongly acidic reagents (diphenylamine, etc.) suffices.

Uranyl Ferrocyanide (1)

Procedure:
Several drops of uranyl acetate solution are placed in a white spot plate. Potassium ferrocyanide is added dropwise until a brown solution results. A drop of the unknown solution to be tested is added. De-colorization indicates the presence of carbonate ions.

Reagents:
0.1grams Uranyl Acetate in 100 mL water
0.1 grams Potassium Ferrocyanide in 100 mL water

Notes: Basic solutions (NaOH, Sodium Sulfide, H_2SO_4) react similarly. Phosphate must be absent. URANIUM SALTS ARE TOXIC.

RETIRED

CHLORATES

Aniline Sulfate (1, 2)

Procedure:
In a white spot plate, a drop of aniline sulfate is added to the test solution. Two drops of concentrated sulfuric acid are added. A blue ring at the aqueous/acid interface indicates the presence of chlorate ions.

Reagents:
5g of Aniline Sulfate in 100 mL water

Notes: Bromates and iodates give purple-blue colors. Color development may not be immediate. Other oxidizing agents may form a pale peach/brown color. Protect the reagent solution from light.

Diphenylamine (1)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. One drop of diphenylamine solution is added, and the formation of a blue color indicates the presence of an oxidizing salt such as chlorate.

Reagents:
1.5g of Diphenylamine and 20mL of H_2SO_4 and 10 mL Acetic Acid.

Notes: MANY oxidizing ions will color the solution blue, including nitrates, nitrites, chlorates and ferric ions. THE REAGENT SOLUTION IS HIGHLY CORROSIVE. Protect the reagent solution from light.

Indigo (8)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. Indigo reagent is added dropwise. At high chlorate concentrations, a yellow color forms which may indicate the presence of chlorate ions.
In dilute solutions, or with the addition of excess indigo reagent, a pale blue color may be retained. Sodium sulfite solution added dropwise may decolorize the blue solution to indicate the presence of chlorate ions.

Reagents:
0.01g Indigo carmine (FD&C Blue #2) in 100 mL concentrated sulfuric acid
5g Sodium Sulfite in 100 mL water

Notes: Indigo blue (D&C Blue #6) reacts similarly, but is less sensitive than Indigo carmine (FD&C Blue #2, also known as soluble Indigo Blue).
Nitrates decolorize the blue indigo reagent initially and also can form a similar yellow solution.

RETIRED

CHLORIDES

Silver Nitrate (1)

Procedure:
One or two drops of the unknown solution is placed in a black spot plate. To this is added a drop of silver nitrate. A white precipitate indicates the presence of chloride ions.

Reagents:
5g of Silver Chloride in 100 mL water
Concentrated Ammonium Hydroxide

Notes: Sulfates may form a white precipitate. As sulfates are insoluble in basic solutions, if the white precipitate re-dissolves in concentrated ammonium hydroxide, it confirms the presence of chloride. Other halides react similarly, although their precipitates vary from off-white to yellow in color.

Since basic solutions forma brown interference, another aliquot of the unknown solution can be acidified with dilute nitric acid and retested. STORE THE SILVER CHLORIDE SOLUTION IN DARKNESS (opaque container).

Take ppt – dissolve in NH_4OH → to dryness microscope slide → octahedron crystals

Reagents:
2g of Oxine (8-Hydroxyquinoline) in 100 mL of 1:4 Acetic Acid
2 parts 6% Hydrogen Peroxide with 1 part 1:4 Acetic Acid
1g Silver Nitrate in 100 mL water
Concentrated Nitric Acid
Notes: The test is specific for chloride in the presence of other halide ions.

NITRATES

Modified Griess/Zn (2, 3)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. To this is added one drop of sulfanilic acid solution, and one drop of napthylethylenediamine solution. (If a pink/red color forms, see nitrites). The colorless solution will turn pink/red with the addition of powdered zinc to indicate the presence of nitrate ions.

Reagents:
1g Sulfanilic acid dissolved with warming in 100 mL of 30% Acetic Acid.

1g N-1-Napthylethylenediamine in 100 mL of 30% Acetic Acid/70% ethanol.

Zinc dust is heated with dilute acetic acid, cold rinsed with dilute acetic acid, then suction filtered, washed with water, and dried.

Notes: See nitrites if a pink color forms before zinc addition. Very basic solutions may shift the solution color towards orange.

RETIRED

RETIRED

Nitron (6, 7)

Procedure:
One or two drops of the unknown solution is placed in a black spot plate. A drop of the nitron solution is added. The formation of thin, needle-like crystals indicates the presence of nitrate ions.

Reagent:
1g Nitron (diphenylenedianilohydrotriazole) dissolved in 20 mL 88% Formic Acid

Notes: Nitron also precipitates perchlorates. With practice, the needle-like crystals from a nitrate reaction can be distinguished from the wider, blade-like crystals from the perchlorate reaction.

Diphenylamine (1)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. One drop of diphenylamine solution is added, and the formation of a blue color indicates the presence of an oxidizing ion such as nitrate.

Reagents:
1mg of Diphenylamine is dissolved in 10 mL of concentrated sulfuric acid.

Notes: MANY oxidizing salts will color the solution blue, including nitrates, nitrites, chlorates and ferric ions. THE REAGENT SOLUTION IS HIGHLY CORROSIVE. Protect the reagent from light.

NITRITES

Modified Griess (1,32)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. To this is added one drop of sulfanilic acid solution, and one drop of napthylethylenediamine. The formation of a pink/red color indicates the presence of nitrite ions. (If the solution remains colorless, see nitrates).

Reagents:
1g Sulfanilic acid dissolved with warming in 100 mL of 30% Acetic Acid.
1g N-1-Napthylethylenediamine in 100 mL of 30% acetic acid/70% ethanol.

Notes: See nitrates if no pink color forms. Very basic solutions may shift the solution color towards orange.

Diphenylamine (1, 3)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. One drop of diphenylamine solution is added. The formation of a blue color indicates the presence of an oxidizing ion such as nitrite.

Reagents:
1mg of Diphenylamine is dissolved in 10 mL of concentrated sulfuric acid.

RETIRED

Notes: MANY oxidizing salts will color the solution blue, including nitrates, nitrites, chlorates and ferric ions. THE REAGENT SOLUTION IS HIGHLY CORROSIVE. Protect the reagent solution from light.

Triphenylselenium chloride (1)

Procedure:
One or two drops of the unknown solution is placed in a black spot plate. To this is added a drop of triphenylselenium chloride solution. Formation of a white opalescence, or clear, needle-like crystals indicate the presence of perchlorate ions. It may take several minutes for the perchlorate crystals to form in a dilute solution. When in doubt, run a blank solution and wait for up to one hour.

Reagents:
Saturated solution of triphenylselenium chloride in water.

Notes: It may take up to an hour for crystals to form. Extract unknowns with warm water, as perchlorate is sparingly soluble in cold water.

Nitron (6, 7)

Procedure:
One or two drops of the unknown solution is placed in a black spot plate. A drop of the nitron solution is added. The formation of blade-like crystals indicates the presence of perchlorate ions.

Reagents:
1g Nitron (diphenylenedianilohydrotriazole) dissolved in 20 mL 88% Formic Acid

Notes: Nitron also precipitates nitrates. With practice, the needle-like crystals from a nitrate reaction can be distinguished from the wider, blade-like crystals from the perchlorate reaction.
Extract unknowns with warm water, as perchlorate is sparingly soluble in cold water.

Methylene Blue (3)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. Two drops of zinc sulfate solution, two drops of potassium nitrate solution, and one drop of methylene blue solution are added. The formation of a purple color indicates the presence of the perchlorate ion.

Reagents:
Saturated aqueous solution of Zinc Sulfate
20g of Potassium Nitrate in 100 mL water
0.03g of Methylene Blue in 100 mL water

Notes: Persulfates react similarly. Extract unknowns with warm water, as perchlorate is sparingly soluble in cold water.

RETIRED

RETIRED

NITROCELLULOSE

Benzoin-Thiobarbituric Acid (Jungreis-66)

(A "Two-Part" Test)

Procedure:

(Part 1): Add one or more granules (at least 2mg total weight) of suspected smokeless powder to a few centigrams of Benzoin in a small test tube (2-3 inches long). Introduce a strip of filter paper moistened with Griess reagent to within one inch (approximately) of the test mixture. Immerse longer end of tube in a 150° (approximately) oil bath and hold for two minutes. A red stain appears on the test paper when NO_2 is present.

(Part 2): To a separate, similar sample in a similar test tube, add a few centigrams of Thiobarbituric Acid and 2 drops of 85% Phosphoric Acid. Heat as described for Part 1 (above). A yellow-orange color appears within 2-3 minutes when NC is present.

Reagents:

Benzoin and Thiobarbituric Acid are slightly colored solids supplied in powder form.

Notes: Part 1 is a test for "Nitro" groups, Part 2 for Cellulose. Avoid prolonged heating in either step; 2-3 minutes should suffice.

Permanganates/Manganous Salts

Filter Paper

Pre-combustion permanganate is recognizable by its purple color in aqueous solutions. A drop of the purple unknown solution is spotted onto filter paper. The change from a purple to a brown/black stain after 30-60 minutes indicates the presence of permanganate ions which have bene reduced to manganese dioxide by the cellulose in the paper.

Notes: Different oxidation states may result in vividly colored solutions of purple, brown or green.

(Although Permanganate is not commonly encountered, the following is reported:)

Ammonium Persulfate/Silver Nitrate (2, 3)

Procedure:
Mix a drop of the unknown solution with a drop of concentrated sulfuric acid in a small crucible. Add a drop of the silver nitrate solution and 2-3 mg of ammonium persulfate, and heat gently. A red/violet color indicates the presence of manganese.

Reagents:
4g of Silver Nitrate in 100 mL water
Ammonium Persulfate

Notes: This test is for post-combustion permanganates. It also can be used for residues of batteries containing MnO_2. The black manganese dioxide powder is oxidized in a 3% solution of hydrogen peroxide and 1 drop of concentrated sulfuric acid, then tested as above.
STORE THE SILVER NITRATE SOLUTION IN DARKNESS (opaque container)

RETIRED

PHOSPHATES

Commercial Test Kit

A Spectrokit is intended for quantitative detection of ortho-Phosphate ions in aqueous solutions. In practice, the blue coloration resulting from the molybdic complex/acid reaction indicates the presence of phosphate without the use of a spectrophotometer. See sources of chemicals.

Reagents:
The kit comes with ammonium molybate, 1 amino-napthol and sulfonic acid reagents pre-prepared.

Notes: Nitrates may interfere; dilution may overcome interferences.

Ammonium Molybdate/Tin (II) Chloride

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. To this is added one drop of ammonium molybdate solution, and then a drop of tin chloride solution. Formation of a blue color indicates the presence of ortho-phosphate ions.

Reagents:
Small amount of (e.g., 1g) Ammonium Molybdate (tetrahydrate) in 20 mL of 4M Nitric Acid

1.1g Tin (II) Chloride (Stannous) in 5ml of 5M Hydrochloric Acid, which is then diluted to 20 mL with water

Notes: The shelf life of both reagents is approximately one month. Addition of a granule of tin metal to the tin solution may retard oxidation.

SULFATES

Barium Chloride (2)

Procedure:
One or two drops of the unknown solution is placed in a black spot plate. One drop of barium chloride solution is added. A white precipitate which is insoluble in acid indicates the presence of sulfate ions.

Reagents:
5g of Barium Chloride in 100 mL water
Concentrated Acetic Acid

Notes: See carbonates. A precipitate of barium sulfate will not re-dissolve in concentrated acetic acid, while a barium carbonate precipitate will.

Sodium Rhodizonate/Barium Chloride (1)

Procedure:
A drop of barium chloride is placed on filter paper followed by a drop of sodium rhodizonate solution. The red fleck is treated with a drop of faintly acid or alkaline test solution. De-colorization of the red barium rhodizonate indicates the presence of sulfate ions.

Reagents:
0.2g Barium Chloride in 100 mL water
0.1g Sodium Rhodizonate

Notes: THE SODIUM RHODIZONATE SOLUTION MUST BE PREPARED FRESH EVERY OTHER DAY. The procedure may also be carried out in a white spot plate. A suspension of colored barium rhodizonate is prepared by bringing together a drop each of barium chloride and sodium rhodizonate solutions. The color is discharged when a drop containing sulfate is added. It is recommended that a blank be run.

RETIRED

SULFIDE

Sodium Azide (1, 2)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. A drop of the brown sodium azide reagent solution is added. De-coloration with the evolution of gas indicates the presence of sulfide ions.

Reagents:
3g Sodium Azide and 1.3g Iodine dissolved in 100 mL water

Notes: Thiosulfates and thiocyanates react similarly. For dilute solutions, look for the evolution of gas bubbles at the base of the depression. Basic solutions may decolorize the azide solution, but they do

not cause the evolution of gas. The solution has a long shelf life. USE EXTREME CAUTION DISPOSING OF BULK AZIDE SOLUTIONS, DO NOT WASH DOWN DRAINS AS HAZARDOUS COPPER AZIDE MAY FORM.

Lead Acetate Test Paper (8)

Procedure:
Add a drop of the unknown solution to lead acetate test paper. A brown-black stain which forms instantly indicates the presence of sulfide ions.

Reagents:
Lead acetate test paper strips are widely available, see sources of chemicals.

Notes: Hydrosulfides react similarly.

Fuchsin (4)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. One drop of pink fuchsin is added; a de-coloration of the solution indicates the presence of sulfide.

Reagents:
2mg Basic Fuchsin in 100 mL water

Notes: Sulfites react similarly.

RETIRED

SYSTEMATIC ANALYSIS OF LOW EXPLOSIVES
CATION AND MISCELLANEOUS SPOT TESTS

ALUMINUM

Acid/Base Solubility (5)

The suspected aluminum is placed in a white spot plate. Aluminum dissolves readily in dilute sodium hydroxide, and also dissolves in concentrated hydrochloric acid. (See the chart summarizing metal solubilities at the end of this section).

Morin (1, 2, 3)

Procedure:
Dissolve the metallic particle in sodium hydroxide in a spot plate depression. Neutralize the solution with dilute acetic acid. Add two drops of morin solution; observe under ultraviolet light. A vivid green fluorescence indicates the presence of aluminum.

Reagents:
Saturated solution of Morin in Methanol

Notes: A basic solution of aluminum shows a yellow rather than green fluorescence (similar to other metals); it has to be neutral to weakly acidic. Excessive acid may inhibit fluorescence. Tin reacts the same as aluminum, but is not commonly used in flash powders (reaction not as brilliant).

ALUMINUM

Nessler (1, 2, 3)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. One or two drops of Nessler reagent are added. An orange/brown precipitate indicates the presence of ammonium ions.

Reagents:
Dissolve 10g mercuric iodide and 5g potassium iodide in 50 mL water. Add a solution of 20g potassium hydroxide in 50 mL water. Let the solution stand for several days. Decant and store the supernatant liquid in a brown bottle. (Pre-made Nessler reagent is commercially available)

Notes: Many organic solvents, including methanol, acetone and ethanol interfere with the Nessler test. Organic solvents should be completely evaporated before this test is performed. Silver, lead and mercury reportedly interfere.

Commercial Test Paper

Procedure:
The test paper is dipped in the unknown solution, and then 10% sodium hydroxide is dropped onto the region. A golden yellow to brown color indicates the presence of ammonium ions.

Reagents:

RETIRED

10% aqueous Sodium Hydroxide solution
Test paper manufactured by Machery, Nagel & Co. in Germany; see sources.

Notes: See manufacturer's specifications for possible interferences.

ANTIMONY (Military)

Although Antimony is not commonly encountered, the following is reported:

Rhodamine B (2, 3)

Procedure:
One or two drops of the unknown solution are placed in a white spot plate. A drop of concentrated hydrochloric acid is added. (Antimony V can now be detected; in order to detect Antimony III a few milligrams of sodium nitrite must also be added). Several drops of a Rhodamine B solution are added; a change from the fluorescent pink/red to a violet color indicates the presence of antimony.

Reagents:
0.01g Rhodamine B, O in 100 mL water
Concentrated hydrochloric acid

Notes: Pre-explosion antimony can be dissolved in concentrated HCl. Sulfides react similarly.

BARIUM (Military)

Sodium Rhodizonate (1, 2)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. One drop of a sodium rhodizonate solution is added, and if a cloudiness forms, one drop of ethanol and one drop of dilute hydrochloric acid is added. The persistence of a red precipitate after acid addition indicates the presence of barium.

Reagents:
0.01g Sodium Rhodizonate in 100 mL water
10% hydrochloric acid
Ethanol

Notes: Other metals form orange salts, and are also soluble in dilute acid. Strontium reacts similarly. THE SODIUM RHODIZONATE SOLUTION MUST BE FRESHLY MADE EVERY OTHER DAY.

CARBON

Visual/Ignition (5)

The black color and appearance, as well as the characteristic burning with afterglow, indicate the presence of carbon.

RETIRED

IRON (Pyrotechnic Devices/Improvised Devices)

Acid Solubility

Intact iron particles are confirmed by ferromagnetism. The suspected iron particles are placed in a white spot plate. Iron dissolves readily in concentrated hydrochloric acid, producing the yellow colored solution commonly encountered in "muriatic" acid. (See chart summarizing metal solubilities at the end of this section).

Potassium Thiocyanate (1)

Procedure:
After dissolution of iron, or in post-blast extracts, a drop of the unknown solution is placed in a white spot plate. A few drops of potassium thiocyanate solution is added. The formation of a red/brown color indicates the presence of iron (III) ions.

Reagents:
1g Potassium Thiocyanate in 100 mL water

Notes: Only Iron III is detected. Phosphates interfere; nitrites may color the solution.

Dipyridyl (1)

Procedure:
After dissolution of iron, or in post-blast extracts, a drop of the unknown solution is placed in a white spot plate. A drop of dipyridyl solution is added. Formation of a pink or red circle indicates the presence of iron (II) ions.

Reagents:
2g (2,2')-Dipyridyl in 95% ethanol

Notes: Only Iron II is detected.

MAGNESIUM

Acid Solubility

Particles of the suspected metal are placed in a white spot plate. Dilute acid solutions (nitric, acetic, sulfuric or hydrochloric acids) will readily dissolve magnesium. See the chart summarizing metal solubilities at the end of this section. The dissolved metal can be tested as below.

Quinalizarin (1, 2, 3)

Procedure:
One or two drops of the unknown solution are placed in a white spot plate. Two drops of quinalizarin solution are added. An orange color appears in acidic conditions. A solution of sodium hydroxide is added dropwise until a change to violet occurs; the addition of one excess drop produces a blue precipitate or coloration.

Reagents:

RETIRED

0.02g of quinalizarin in 100 mL ethanol

Notes: Run a water blank concurrently in order to accurately recognize any change from violet to blue. Add the sodium hydroxide to excess if in doubt.

PHOSPHOROUS

Oxidize to Phosphate

Detect phosphorus using the phosphate tests. Phosphorus can be burned or placed for 20 minutes in a 1:1 solution of concentrated nitric and hydrochloric acids to obtain phosphate.

POTASSIUM

Commercial Test Paper

Procedure:
Apply a drop of the neutral test solution to the orange test paper, then apply dilute nitric acid to the strip. Upon addition of acid, the paper turns lemon yellow while the spots where the potassium ions have been placed remain orange.

Reagents:
10% aqueous nitric acid
Test Strips manufactured by Macherey, Nagel & Co. of Germany; see sources.

Notes: Ammonium reacts similarly at high concentrations. The test papers may utilize the dipicrylamine reagent method (1, 3).

Zinc Uranyl Acetate (1)

See the sodium test below; concentrated potassium solutions may be identified by a microcrystalline test.

SODIUM

Zinc Uranyl Acetate (1)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. Add one drop of zinc uranyl acetate solution; observe under ultraviolet light. A bright green fluorescence indicates the presence of sodium ions.

Reagents:
A. 10g Uranyl Acetate dissolved in 50 mL 30% Acetic Acid
B. 30g Zinc Acetate stirred with 3 mL 30% Acetic Acid. Solution B diluted to 50 mL with distilled water.

RETIRED

Solutions A and B are warmed and then mixed warm. Heat gently until a clear solution results. Add a trace of sodium chloride, stopper, and let stand for 24 hours. Filter off the precipitated zinc uranyl acetate and discard. Store the filtrate in a glass stoppered bottle.

Notes: This reagent has a long shelf life. In dilute solutions, run blank reagent in the depression next to the test solution for comparison. Concentrated solutions of potassium can interfere. If the solution is very concentrated, a microcrystalline discrimination is possible between sodium (triangular) and potassium (needle-like) crystals.

RETIRED

STRONTIUM

Flame Test (4)

A particle of the suspected material is placed on a platinum wire and inserted into an open flame. The presence of a crimson red color indicates the presence of strontium ions.

Sodium Rhodizonate (1, 2)

See Barium; Strontium reacts similarly.

SUGAR

Naphthol (2, 3)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. Add one drop of naphthol solution and two drops of concentrated sulfuric acid. The appearance of a blue or purple-blue color indicates the presence of sugar.

Reagents:
15g of 1-Naphthol in 100 mL Ethanol

Notes: Fructose forms a similar purple-blue color. Glucose and maltose form pink colors at the sulfuric acid interface. Keep reagent from light.

SULFUR

Pyridine/NaOH (3, 4, 5)

Procedure:

Place a yellow piece of suspected sulfur in a small test tube or shell vial. Add a few drops of pyridine and gently warm. Remove from heat and add two drops of sodium hydroxide solution. The formation of a yellow, blue or brown color in the aqueous layer indicates the presence of sulfur.

If no particles of sulfur are visible, wash the dry solid with pyridine, collect the filtrate and test with sodium hydroxide as above. A disposable glass pipet with glass wool plugs can be used as a filtering mechanism.

Reagents:
Pyridine
10g Sodium Hydroxide in 100 mL water

Notes: Water interferes. USE CAUTION (and a fume hood) WITH PYRIDINE.

Benzoin/Lead Acetate Test Paper (1)

Procedure:
A small quantity of dry unknown powder is placed in a small test tube or shell vial with an equal quantity of benzoin. The tube's mouth is closed with moist lead acetate paper; the tube is then warmed. A brown/black staining of the test paper indicates the presence of sulfur.

Reagents:
Lead acetate test paper strips are widely available; see sources.

Notes: An oil bath at 150° is recommended; if using a hot plate or micro-burner, heat the melted benzoin for approximately 30 seconds. This test works on intact pre-combustion mixtures containing sulfur.

ZINC

Acid Solubility

Particles of the suspected zinc metal are placed in a white spot plate. Zinc is soluble in concentrated hydrochloric, nitric and sulfuric acids. Zinc is sparingly soluble in 10% sodium hydroxide and not very readily soluble in 10% acids. See the chart summarizing metal solubility below.

Dithizone (1, 2, 3)

Procedure:
One or two drops of the unknown solution is placed in a white spot plate. A drop of sodium hydroxide solution is added followed by a drop of dithizone solution. If the green dithizone turns red, it indicates the presence of zinc ions.

RETIRED

SUMMARY OF METAL SOLUBILITIES

CONCENTRATED ACIDS					DILUTE ACIDS (10%)				Dilute NaOH (10%)
H_2SO_4	HNO_3	HCl	Acetic		H_2SO_4	HNO_3	HCl	Acetic	
-	-	+	-		-	-	slow +	-	+
Slow +	Very slow	YELLOW +	-		-	+	slight +	-	-
+	Fume +!	+	+		+	+	+	+	–
-	-	-	-		-	-	-	-	-
Slow +	Fume +!	+	Slow +		Slow +	+	-	-	Slow +

RETIRED

SOURCES OF CHEMICALS
SYSTEMIC ANALYSIS OF LOW EXPLOSIVES
SPOT TESTS

Most chemicals are readily available from major suppliers of chemicals.

Many chemicals are available in conveniently small size from Chem Services, Inc. West Chester, PA (215) 692-3026

Some specialty chemicals are available from KODAK Laboratory and Research products, Rochester, NY 14650, (800) 225-5352

Triphenylselenium chloride is available from PFALTZ AND BAUER, Division of Aceto Chemical Co., Inc., 172 E. Aurora St., Waterbury, CT 06708 (203) 574-0075

Milton Roy (formerly Bausch and Lomb) manufactures SpectroKits for the detection of phosphate, chloride, sulfate, sulfite, and other ions. Milton Roy, Analytical Systems Division, 820 Linden Ave., Rochester, NY 14625, (716) 248-4000

A wide variety of test papers, including those for ammonium and potassium, are marketed by GALLARD/SCHLESINGER Chemicals and Research Products, 584 Mineola Ave., Carle Place, New York 11514 (516) 333-5600.

The above is for informational use only; not intended as endorsement.

ABBREVIATED REFERENCES FOR SYSTEMATIC ANALYSIS OF LOW EXPLOSIVES

SPOT TESTS:

1) Feigl, Anger, Spot Tests in Inorganic Analysis, Elsevier, NY 10017, 6th Ed 1972.
2) General Information Bulletin 74-8, National Bomb Data Center, 1974
3) Jungries, Spot Test Analysis – Clinical, Environmental, Forensic and Geochemical Applications, Chemical Analysis, vol 75, 1985.
4) Lange, Handbook of Chemistry, McGraw Hill, 10th Ed., 1971.
5) Meyers, "A Systematic Approach to the Forensic Examination of Flash Powders", Journal of Forensic Sciences, Vol. 23, #1, 1978.
6) Parker, Stephen, McOwen, Cherolis, "Analysis of Explosives and Explosive Residues, Part 1: Chemical Tests," Journal of Forensic Sciences, Vol. 20, #1, 1975.
7) Scott's Standard Methods of Chemical Analysis, (Furman Editor), Van Nostrand, NY, 6th Ed., 1962.
8) Svehla, Vogel's Qualitative Inorganic Analysis, 6th Ed., Wiley, 1987.

APPLICATIONS OF SPOT TESTS TO EXPLOSIVES ANALYSIS

Hoffman and Byall, "Identification of Explosive Residues in Bomb Scene Investigations," Journal of Forensic Sciences, Vol. 19 #1, 1974.

RETIRED

Washington, Kopek, Midkiff, "Systematic Approach to the Detection of Explosive Residues, V, Black Powders", Journal of the Association of Official Analytical Chemists, Vol. 60, November 1977.

RETIRED