APPENDIX A - SPOT TEST PROCEDURES (ATF)

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SYSTEMATIC ANALYSIS OF LOW EXPLOSIVES

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SYSTEMATIC ANALYSIS OF LOW EXPLOSIVES - ANION SPOT TESTS

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

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	-	Pb, Zn**	Sol base, acid
	Morin	-	Tin, Basic **	UV green fluor.
Ammonium	Nessler	0.01% NH_4 OH		Orange/brown
	Commercial Test Paper	0.01% "	*	Brown
Antimony	Rhodamine B		Sulfide*	Violet
Barium	Sodium Rhodizinate	0.1mg $BaNO_3$	Sr, FRESH RGT!*	Red ppt.
Carbon	Visual/Ignition	-		Black/ glowing
Iron	Acid Solubility	-	**	Sol HCl, yellow
	Potassium Thiocyanate	-	PO_4 NO_3 *	Red ppt(Fe III)
	Dipyridyl	-		Pink/Red (FeII)
Magnesium	Acid Solubility	-	**	Sol dilute acid
	Quinalizarin	-		Change to blue
Phosphorus	Oxidize to Phosphate	-		See phosphate
Potassium	Commercial Test Paper	0.01mg KNO_3	Ammonium	Orange
	Zinc Uranyl Acetate	>1.0mg "	Sodium	Needle crystals
Sodium	Zinc Uranyl Acetate	0.1mg $NaNO_3$	Conc. Potassium	UV green fluor.
Strontium	Flame Test			Crimson red
	Sodium Rhodizonate	0.1mg $SrNO_3$ 2	Ba, FRESH RGT!	Red ppt.
Sugar	Napthol	0.1mg Sugar	(others pink)	Purple
Sulfur	Pyridine/NaOH	0.001mg S	Water, contam.	Ylw/blue/brown
	Benzoin/Lead Acetate	-		
Zinc	Acid Solubility	-	**	Sol conc acid
	Dithizone	-		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, pshosphoric 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.

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:
5 g of Barium Chloride in 100 mls 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. Decolorization indicates the presence of carbonate ions.

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

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

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:
~~5 g of Aniline Sulfate in 100 mls 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:
1.5 g DPA + 20 ml H_2SO_4 + 10 acetic acid
~~1 mg of Diphenylamine is dissolved in 10 mls of concentrated sulfuric 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:
0.01 g Indigo carmine (FD&C Blue #2) in 100 mls concentrated sulfuric acid
5 g Sodium Sulfite in 100 mls 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.

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:
5 grams of Silver Chloride in 100 mls water
Concentrated Ammonium Hydroxide

Notes: Sulfates may form a white precipitate. As sulfates are insoluble in basic solutions, if the white precipitate redissolves 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 form a 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).

Allow ppt in lite turn gray

TAKE PPT - dissolve in NH4OH = 4 tiny needles microscope
Side = 4 octahedron crystals

Oxine/Silver Nitrate (1)

Procedure:
One or two drops of the unknown solution is placed in a small test tube with a drop of the oxine solution, a drop of the hydrogen peroxide solution and a microdrop of nitric acid. The mixture is warmed for approximately four minutes. A drop of silver nitrate solution is then added. The appearance of turbidity or a colorless precipitatey indicates the presence of chloride ions.

Reagents:
2 grams of Oxine (8-Hydroxyquinoline) in 100 mls of 1:4 Acetic Acid
2 parts 6% Hydrogen Peroxide with 1 part 1:4 Acetic Acid
1 g Silver Nitrate in 100 mls 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 naphthylethylenediamine 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:
1 g Sulfanilic acid dissolved with warming in 100mls of 30% acetic acid.
1g N-1-Napthylethylenediamine in 100 mls 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.

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:
1 g Nitron (diphenylenedianilohydrotriazole) dissolved in 20mls 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:
1 mg of Diphenylamine is dissolved in 10 mls 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, 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. The formation of a pink/red color indicates the presence of nitrite ions. (If the solution remains colorless, see nitrates).

Reagents:
1 g Sulfanilic acid dissolved with warming in 100mls of 30% acetic acid.
1g N-1-Napthylethylenediamine in 100 mls 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, and the formation of a blue color indicates the presence of an oxidizing ion such as nitrite.

Reagents:
1 mg of Diphenylamine is dissolved in 10 mls 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 solution from light.

Perchlorate.

Sat. Cesium sulfate + Ammonium hydroxide = ppt.

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 it 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.

Reagent:
1 g Nitron (diphenylenedianilohydrotriazole) dissolved in 20mls 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
20 g of Potassium Nitrate in 100 mls water
0.03 g of Methylene Blue in 100 mls water

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

[redacted]ROCELLULOSE

BENZOIN-THIOBARBITURIC ACID (Jungreis-66)

(A "Two-Part" Test)

Procedure:

(Part 1): Add one or more granules (at least 2 mg. total wt.) 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 2 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 [redacted] is present.

NC

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.

[redacted]/Manganous Salts

Filter paper

Precombustion permanganate is recognizible 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 been 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 mls of 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)

PHOSPHATES

Commercial Test Kit

A Spectrokit is intended for quantitative detection of ortho-Phosphate ions in aqeuous 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 molybdate, 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.

[redacted]

[redacted] g Ammonium Molybdate (tetrahydrate) in 20 mls of 4 M Nitric Acid
[redacted] g Tin (II) Chloride (Stannous) in 5 mls of 5 M Hydrochloric acid
which is then diluted to 20 mls 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:
5 g of Barium Chloride in 100 mls of water
Concentrated Acetic Acid

Notes: See carbonates. A precipitate of barium sulfate will not redissolve 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 followied by a drop of sodium rhodizonate solution. The red fleck is treated with a drop of faintly acid or alkaline test solution. Decolorization of the red barium rhodizonate indicates the presence of sulfate ions.

Reagents:
0.2 g Barium Chloride in 100 mls water
0.1 g 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.

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. Decoloration with the evolution of gas indicates the presence of sulfide ions.

Reagents:
3 g Sodium Azide and 1.3 g Iodine dissolved in 100 mls 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 decoloration of the solution indicates the presence of sulfide.

Reagents: 2 mg Basic Fuchsin in 100 mls water

Notes: Sulfites react similarly.

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, and observe under ultraviolet light. A vivid green fluoresence 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 brillant

AMMONIUM

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 10 g mercuric iodide and 5 g potassium iodide in 50mls of water. Add a solution of 20g potassium hydroxide in 50 mls 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:
10 % aqueous Sodium Hydroxide solution
Test paper manufactured by Machery, Nagel and Co in West Germany, see sources.

Notes: See manufacturers specifications for possible interferences.

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

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:
0.01 g Rhodamine B , 0 in 100 mls 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 persistance of a red precipitate after acid addition indicates the presence of barium.

Reagents:
0.1 g of Sodium Rhodizonate in 100 mls of 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.

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 of Potassium Thiocyanate in 100 mls 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:
2 g (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; then the addition of one excess drop produces a blue precipitate or coloration.

Reagents:
0.02 g of quinalizarin in 100 mls 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.

PHOSPHORUS

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;