

## TECHNICAL NOTE

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### A Systematic Approach to the Forensic Examination of Flash Powders

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Flash powders are classified as detonating pyrotechnic mixtures that deflagrate when unconfined and detonate when confined, emitting an intense light under both conditions. Their legitimate uses include commercial fireworks and military ordnance and simulators. The relative ease of acquiring the chemicals needed to manufacture flash powder has made these mixtures attractive to the amateur bomber. Approximately 15% of the several hundred bomb cases processed annually by this laboratory involve the use of flash powder mixtures.

The examination and identification of some components of low explosives have been briefly described in the literature [1-5], but these articles do not deal specifically with flash powders nor do they describe a systematized approach to their analysis. This paper describes a systematic approach to the examination and identification of gram quantities of flash powder mixtures. The same procedure can easily be adapted to smaller quantities of material such as those found in bomb residues.

#### **Background**

In all flash powder formulations at least one reducing agent and one oxidizing agent are present. Sometimes a tinder material will be added to modify the burning characteristics. Aluminum is the most common reducing agent. It is also the component that produces the bright light associated with fireworks displays. Magnesium can be used as a reducing agent, but it is not very suitable since it is less stable than aluminum toward moisture. Pyrotechnic mixtures containing magnesium have poor stability unless the metal particles are first coated with a protective layer of linseed oil or similar hydrophobic material. Aluminum is most often coated to prevent any reaction within the flash powder formulation.

While aluminum is found in a few dynamite formulations and in water-gels, these explosives will not be confused with flash powder mixtures by the forensic analyst because the oxidizers used in flash powder mixtures are different from those present in other explosives containing aluminum. The analyst should therefore identify at least one oxidizing agent in the residue to differentiate flash powders from dynamites. Potassium chlorate,

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potassium perchlorate, potassium nitrate, and sodium nitrate are commonly used oxidizing agents in flash powders. Cost/performance criteria dictate which of these are used in any one commercial formulation.

Carbon, sulfur, and antimony sulfide are frequently used as tinders. Tinders are easily ignitable materials whose heat of combustion facilitates positive ignition of the pyrotechnic fuel. Many commercial flash powders contain wood granules as tinder and no sulfur; this permits their differentiation from improvised mixtures.

Some commonly encountered flash powder formulations appear in Table 1. The indicated

TABLE 1—*Typical flash powder formulations.*

Formulation	Components	Approximate Weight, %
1	aluminum	40
	sulfur	5
	potassium perchlorate	55
2	aluminum	40
	sulfur	5
	potassium chlorate	55
3	aluminum	35
	potassium perchlorate	65
4	aluminum	30
	potassium chlorate	70
5	carbon	15
	aluminum	15
	sulfur	5
	potassium perchlorate	65
6	carbon	15
	aluminum	15
	sulfur	5
	potassium chlorate	65
7	carbon	15
	aluminum	15
	sulfur	5
	potassium nitrate	65
8	carbon	13
	aluminum	20
	potassium perchlorate	67
9	carbon	15
	aluminum	20
	potassium chlorate	65
10	aluminum	25
	sulfur	5
	potassium perchlorate	50
	antimony trisulfide	20

percentages of each component are rough averages. In our experience substantial variations are found in both commercial and improvised products. The most commonly encountered flash powder formulations are 1 and 2, containing aluminum, sulfur, and potassium perchlorate or potassium chlorate.

#### Analytical Procedure

The procedure shown in the flow diagram (Fig. 1) has been successfully used in our laboratory to examine suspected flash powders. This systematic approach permits identi-

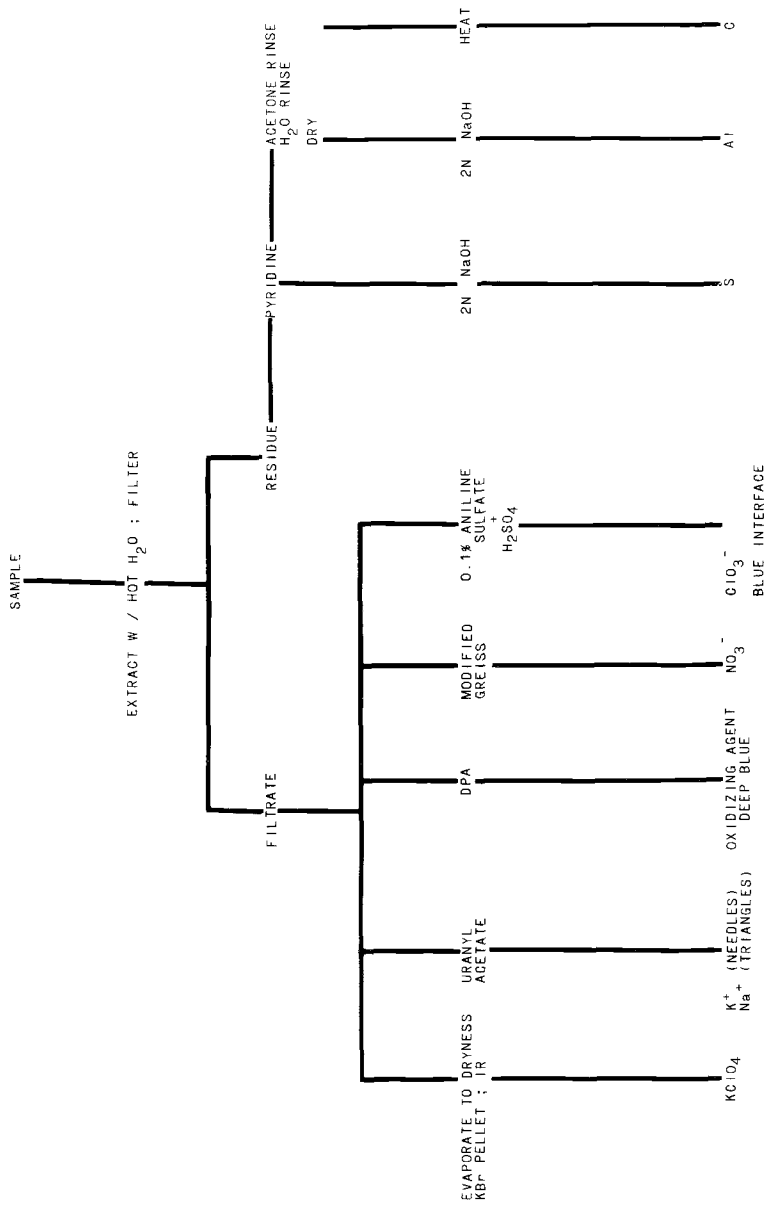


FIG. 1—Flow diagram for the analysis of flash powders.

fication of all components commonly found in flash powder mixtures. Although the procedure describes the analysis of gram quantities of suspected flash powders, the method can be easily adapted to the examination of trace quantities.

Approximately 1 g of sample is analyzed for aluminum, sulfur, charcoal, potassium perchlorate, potassium chlorate, potassium nitrate, and sodium nitrate. Approximately 30 ml of hot water is added to the sample in a 100-ml beaker. The sample is then heated approximately 10 min and filtered; the filtrate is then collected. The material is further extracted on the filter paper with approximately 70 ml of hot water. The combined filtrates contain any potassium perchlorate, potassium chlorate, potassium nitrate, or sodium nitrate present in the mixture. Aluminum, sulfur, or carbon will remain as residue on the filter paper.

Fifteen millilitres of the hot filtrate is removed and retained. The remaining portion is evaporated to dryness. Some of the residue is scraped out and analyzed by infrared spectroscopy with the KBr pellet technique. Figure 2 is a KBr pellet spectrum of potassium perchlorate; Fig. 3, of potassium chlorate; and Fig. 4, a mixture containing potassium perchlorate, potassium chlorate, and potassium nitrate.

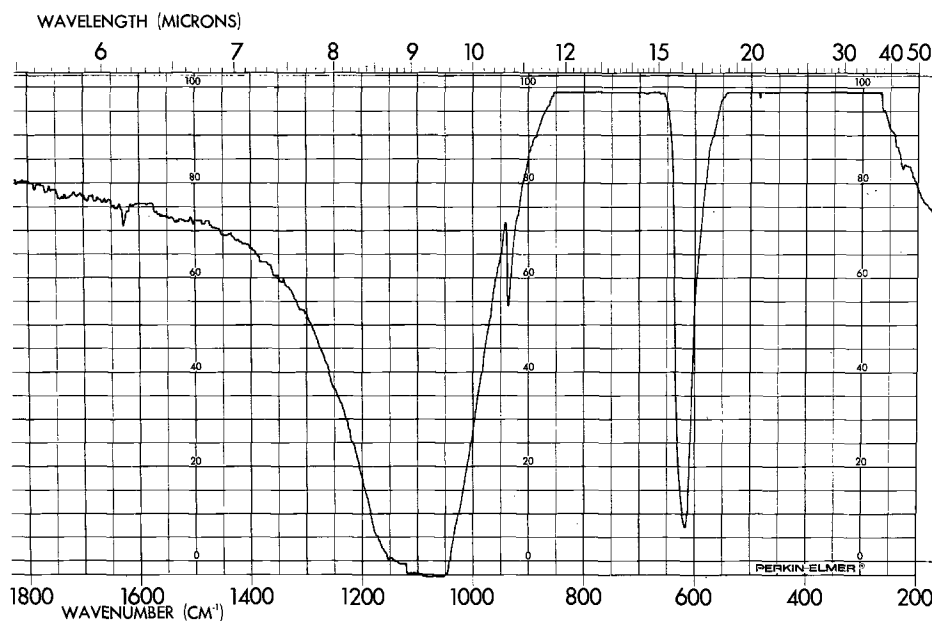


FIG. 2.—Spectrum (KBr pellet) of potassium perchlorate.

Five microlitres of the second portion of filtrate is allowed to dry on a microscope slide. A coverslip is placed over the sample, and two drops of uranyl acetate are allowed to run under the coverslip. The preparation is examined with a transmitted light microscope set at low magnification ( $\times 30$  to  $\times 100$ ). If potassium ions are present, needle-shaped crystals will be observed. If sodium ions are present, triangular-shaped crystals will be observed. Figures 5 and 6 are photomicrographs of potassium uranyl acetate and sodium uranyl acetate, respectively.

Two drops of diphenylamine solution are added to two drops of the filtrate in a white spot plate. A deep blue color indicates the presence of an oxidizing agent.

To test for nitrates, two drops of *N*-(1-naphthyl)ethylenediamine reagent and two drops of sulfanilic acid reagent are added to two drops of the filtrate on a white spot plate.

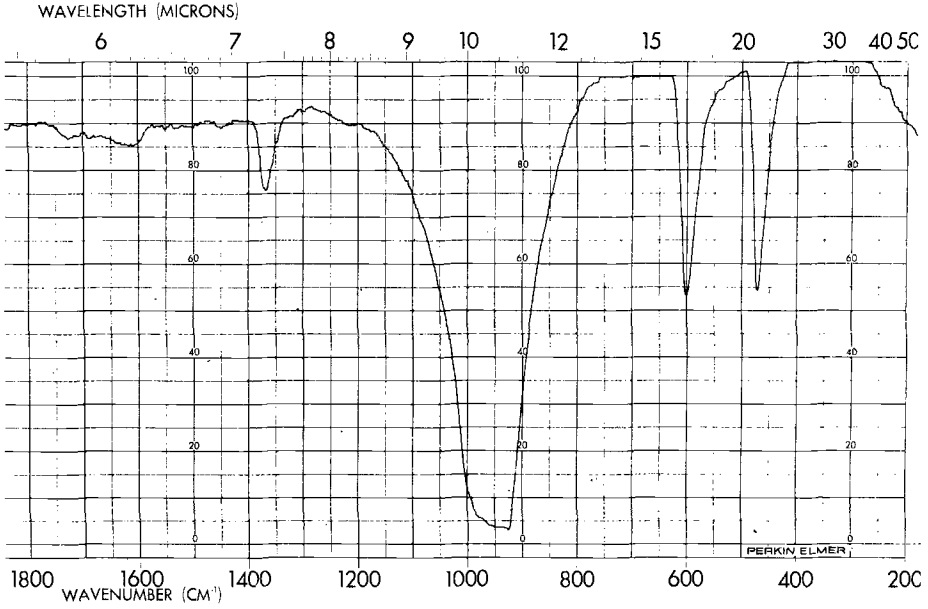


FIG. 3—Spectrum (KBr pellet) of potassium chlorate.

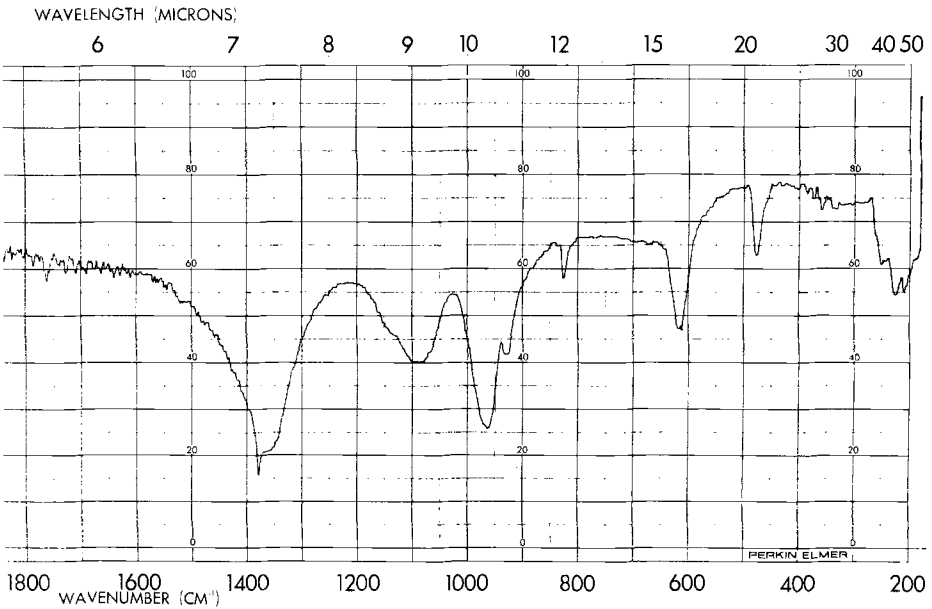


FIG. 4—Spectrum (KBr pellet) of a mixture of potassium perchlorate, potassium chlorate, and potassium nitrate.

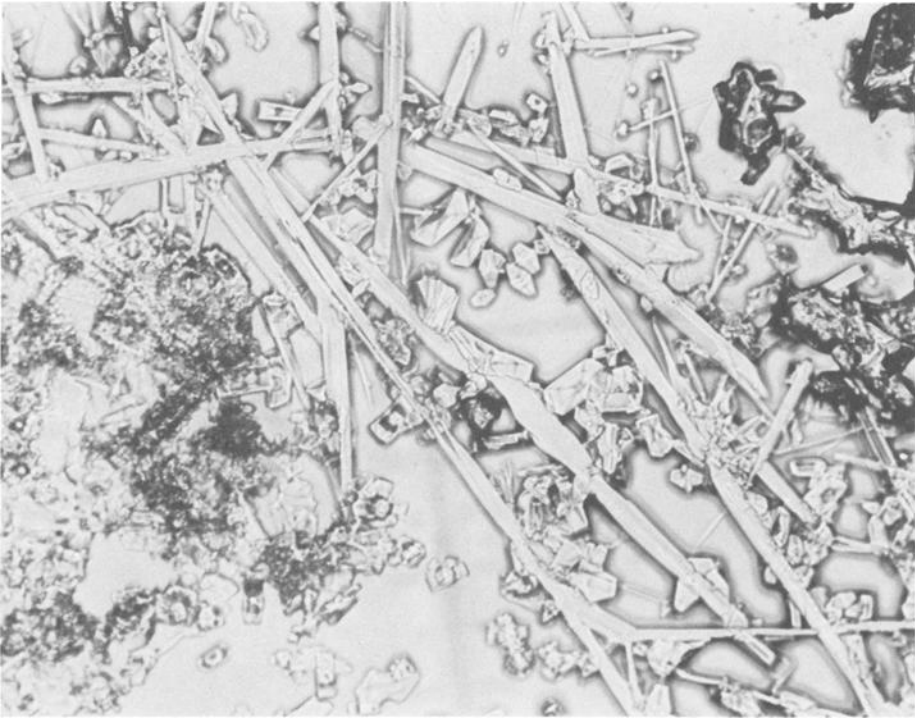


FIG. 5—Photomicrograph of *potassium uranyl acetate*.

Then a small quantity of zinc dust is added. The appearance of a deep red-rose color after the addition of zinc dust indicates the presence of nitrates [3].

Six drops of the hot-water filtrate are placed in a small test tube or glass vial. Eight drops of 0.1% aniline sulfate are added, and the tube is agitated to mix the solutions. Next, 15 drops of concentrated sulfuric acid are added cautiously, the drops being allowed to run slowly down the inside of the tube. If chlorates are present a blue-violet color forms at the interface of the water-acid layer within 1 min.

The filter paper containing the solid residues is dried. The residue is extracted with pyridine to remove any sulfur that may be present. The presence of sulfur in the pyridine filtrate is shown by the appearance of a blue to brown color upon the addition of a few drops (3 or 4) of 2*N* sodium hydroxide. The observed color depends on the concentration of sulfur present: blue for low concentrations, brown for high concentrations.

The filter paper and residue are washed with acetone to remove any residual pyridine, and the filter paper is rinsed with water. The filter paper containing the solid residues is then dried, and a small portion of any solid residue present is placed on a platinum crucible cover. The cover is heated to a red heat, and the characteristic afterglow of carbon is observed when the cover is removed from the flame.

A small quantity of residue from the filter paper is placed on a microscope slide, and two drops of 2*N* sodium hydroxide are added. Gas (hydrogen) evolution indicates the presence of aluminum metal.

One half gram of the original sample is dissolved in 1 ml of cold distilled water, and two drops of this solution are placed on a white spot plate. A crystal of sodium nitrite is added, and the solution is made acidic with a drop of 1:1 hydrochloric acid. When effe-

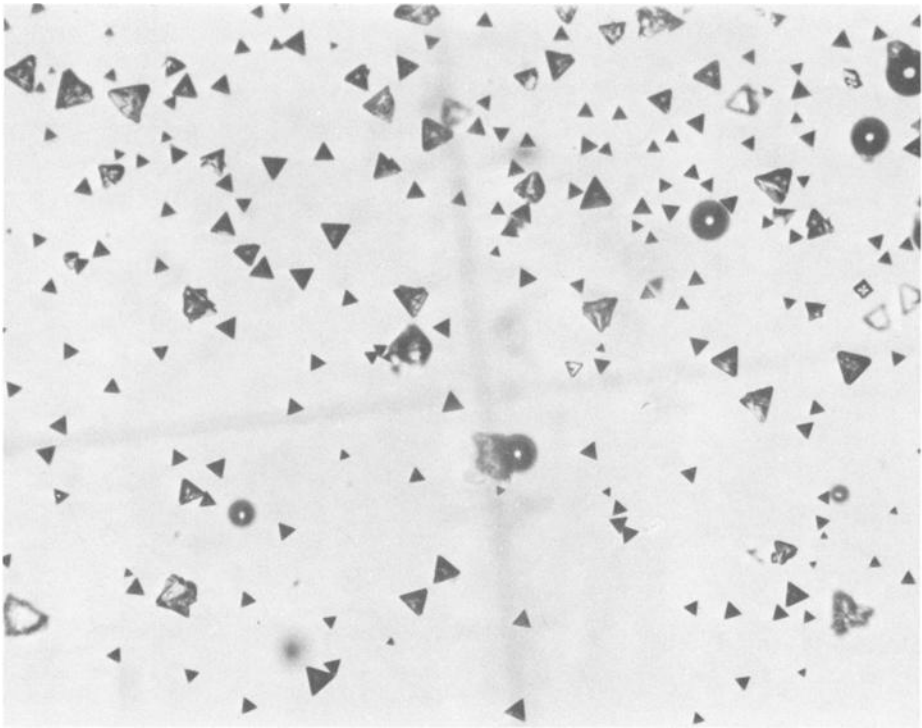


FIG. 6—Photomicrograph of sodium uranyl acetate.

vescence ceases, two drops of a 0.1% aqueous solution of rhodamine B is added. A change from red to violet occurs when antimony is present.

Two drops of sodium azide-iodine solution are added to 0.1 g of the original sample on a white spot plate. A color change from brown to colorless with the evolution of gas (nitrogen) indicates the presence of sulfides.

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## APPENDIX

### **Chemical Reagents**

The methods of preparation of several reagents (analytical reagent grade) commonly used in testing flash powders are listed below.

***N*-(1-Naphthyl)ethylenediamine**/Dissolve 0.5 g *N*-(1-naphthyl)ethylenediamine in 100 ml of methyl alcohol.

**Aniline Sulfate**/Add 1 part of aniline to 1000 parts of distilled water. Add concentrated sulfuric acid by drops to the mixture, with stirring, until the aniline dissolves.

**Diphenylamine**/Dissolve 1 g of diphenylamine in 100 ml of concentrated sulfuric acid.

**0.1% Rhodamine B**/Dissolve 0.1 g of rhodamine B in 100 ml of distilled water.

**Sodium Azide-Iodine**/Dissolve 1 g sodium azide, 1 g potassium iodide, plus a tiny crystal of iodine, in 3 ml water.

**2N Sodium Hydroxide**/Dissolve 80 g of sodium hydroxide in 150 to 200 ml of water and dilute to 1 litre.

**Sulfanilic Acid**/Dissolve 1 g of sulfanilic acid in 100 ml of 30% acetic acid.

**Uranyl Acetate**/Dissolve 2 g of uranyl acetate in 5 ml of warm acetic acid. Add 15 ml water and filter.

### References

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