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Field Spot-Test Kit for Explosives

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ABSTRACT

A spot-test kit was developed to identify the presence of explosives as contaminants on equipment and materials in processing buildings in explosives areas. Three reagents in a carrying case with all the tools needed, including a portable ultraviolet (UV) lamp, can be used in the field to detect any of the common explosives used at Los Alamos. The test is simple. After a suspect area or material is wiped with a clean filter paper, a drop of each of the three reagents placed on different parts of the sample will change color when explosives and/or other nitro compounds are present. A UV light (short wavelength, 254 nm) enhances color for RDX/HMX explosives. If the sample cannot be identified it is collected for laboratory analysis. The current procedure, which works well at Los Alamos, is included as an appendix.

I. INTRODUCTION

In the interest of safety in processing buildings where explosive dusts settle on machinery, fixtures, and materials, it is necessary to know if this residue constitutes a hazard should operations be needed to move the materials and fixtures or if maintenance of piping and utilities is needed. Work can be initiated only when the area is free of explosives. If explosives are present, the articles in question have to be cleaned before any operation starts. A certifying agent makes these judgments using a spot-test kit for explosives detection developed in our analytical laboratory. The spot-test kits are also used to detect and estimate contamination of explosive dusts in soil and shrapnel and to determine if explosives are present in spent hydraulic fluids.

This report describes the tests, the reagents, utensils, and the fitted kit needed to make this test credible. The appendix gives a detailed operating procedure based on Los Alamos standing operating procedures (SOPs) and analytical instructions (AI).

II. DISCUSSION

Spot tests are based on the work of Feigl,[1] who describes methods of using a drop of a reagent on a milligram or less sample that, when reacted, will change color and identify a compound. The drop method uses spot plates or filter paper as the reaction vessels. In the chemistry of explosives, many explosives will react with alkali solutions to form complex colored compounds, and these reactions are used to develop tests for the presence of explosives. Amine compounds also react with compounds containing nitro groups to give colored reactions. The earlier techniques here used sodium hydroxide in solutions of acetone, ethanol, and methanol, and solutions of diphenylbenzidine in concentrated sulfuric acid. They worked but required much care. A paper by Malotky and Downes[2] describes a kit for field use that claims to be specific for different explosives. The application of their method to our operations is not appropriate, as only the presence or absence of explosives needs to be known here. Identification of any explosive, if needed, is done by the on-site explosives analytical laboratory using state-of-the-art techniques. A third reference by Jungreis[3] gives tests for specific explosives.

Before any drop of reagent can be applied to a sample, the sample needs to be gathered onto a matrix or into a container. Scooping a sample with a spatula and placing it on a white ceramic spot plate or onto white plastic bottle caps is one way. Another way is to take a clean filter paper and swipe the area in question to gather enough sample so that, when a drop of reagent is added, the paper acts as a paper chromatogram: then the explosive is dissolved or dispersed through the paper and reacted with the reagent. The dark color on a white background makes it easy to semiquantify the explosive contamination. These sampling methods are described by Feigl[1] and work very satisfactorily in the field.

Before the reagents were incorporated into a test kit, extensive testing was done on the common explosives (TNT, Comp B, RDX, HMX, PETN, tetryl) used by Los Alamos. Ten laboratory technicians correctly identified the six pure explosives present with no difficulty. In addition, 14 other samples, including inerts and nitro compounds, were used as test materials. The 10 technicians picked out the explosives readily and had few doubts about the inerts and nitro compounds. These results prompted us to adapt the system to field use. The test kit went through several changes, from being packed in a wood case to the present broad-base-metal case that contains all the material to make a test and deliver a sample in a package to the analytical laboratory for further analysis.

Because amine/solvent reagents produce color changes, a search was made for a combination of two solvents that would give a dark color quickly. After many combinations, a mixture of N,N-dimethylformamide and di-n-butylamine, 80%/20% by volume, gave the sharpest color change with TNT and similar explosives. This reagent was denoted as "Reagent A." This reagent will detect TNT and other nitrotoluenes, with the color of the TNT reaction predominating. If a black-to-purple color shows on the test, no further tests are necessary. The article in question needs to be cleaned and retested before it can be moved or worked on.

For checking soil contaminated with TNT, it was possible to detect a content as low as 0.01% (100 ppm) as determined by laboratory experiments. Controlled samples of 0.5%, 1.0%, and 1.5% by weight of explosive in soil gave a reaction that a trained technician could easily distinguish quantitatively. Samples with 10%, 20%, 30%, and 40% by weight of explosive gave proportionally darker colors that were qualitatively discernible. This test is strictly qualitative, and no attempts should be made to assign a quantitative value.

The second reagent in the kit is a dilute solution of tetra-n-butyl-ammonium hydroxide in methanol (E-7774) with dichlorofluorescein in a carrier of dimethyl sulfoxide (DMSO), 2%/98% by volume. This is "Reagent B" and its preparation is given in the appendix. The ultraviolet (UV) lamp (254-nm wavelength) defines the presence of RDX, HMX, and PETN. Again, a drop of Reagent B on a spot plate containing the sample or on a filter paper will give a characteristic color change for the explosive sample. When the UV lamp is shined on the filter paper, the RDX, HMX, and PETN will turn blue-black, and after a while the dissolved explosives will migrate and show feathering on the filter paper. A blank filter with a drop of Reagent B will glow with a yellow color. If any explosive is present, a blue-black color is seen easily as the fluorescent glow is blanked out, giving excellent contrast. A short-wavelength UV lamp is used because a long-wavelength UV light causes such a strong fluorescence of the indicator that other colors are obliterated. DMSO is a good solvent for most explosives and will migrate on the filter paper, concentrating the solution so the color reactions can take place and be enhanced with UV light. Experience by skilled operators shows that this reagent and UV lamp make a reliable test in the field.

The third reagent, to identify the presence of triaminotrinitrobenzene (TATB), an insensitive high explosive currently used at Los Alamos, is based on work by B. W. Harris. The TATB is not soluble in common solvents and will not react with the previously described two reagents. The developmental work was reported by B. W. Harris in a paper[4] and was awarded a patent.[5] The solution is 5 g of potassium hydroxide (KOH) in 5 ml of water, all dispersed in 90 ml of DMSO. This was designated "Reagent C" in the test kit. A drop of reagent is placed on a filter paper containing the sample swiped from a source. After a few minutes, the TATB reacts with the reagent and will generate a red color. The addition of UV light will enhance the red color. Usually TATB is mixed with a binder and in this combination will also give a red color. If other explosives are mixed with TATB, the previous two reagents will give a positive test and will mask this test. To detect TATB, the field technician needs to wash out the other explosives with acetone and apply Reagent C to the residue. Because TATB is a relatively new material used in processing at Los Alamos, its presence as a contaminant will be minimal because of the stringent containment practices for waste disposal at the operating sites.

The uses for this kit have expanded beyond our original intentions. In the current environmental testing, the screening of hundreds of soil samples is rapid for picking those that show explosives present. When the test is positive, a sample is sealed in a plastic envelope and delivered to the explosives analytical laboratory for a thorough analysis. In one series of samples, a field technician picked out a dozen worst-case samples out of a hundred. In addition, a clean sample was included. After Soxhlet extraction and quantification by high-performance liquid chromatography (HPLC) methods, the laboratory results verified the technician's rough estimates.

Another use for this spot test is to detect explosives in fuel oil and spent hydraulic fluid. If the spot test indicates the presence of explosives, HPLC or thin layer chromatography will then verify the presence or absence.

When wet samples are swiped on filter paper and allowed to dry, the tests work just as well. If there is doubt in the field technician's judgment, because a chemical other than explosive gives a positive indication, the sample can be sent to the analytical laboratory for extraction and verification by other methods. If the test gives a positive indication and there is some uncertainty, and if the laboratory test is negative, this error on the positive side is not necessarily detrimental.

This kit contains only organic solvents, as the concentrated acids have been removed from previous versions. Although the organic chemicals have been documented in Material Safety Data Sheets (MSDS), the use of a drop per test minimizes any possible hazards. Each technician is made aware and reads the MSDS.

As new and alternate certifiers need to use this spot test, they are trained by Staff Members at the Group M-1 Analytical Laboratory. They are given hands-on experience and a test to verify their judgment before they are authorized for certifying work. These procedures are outlined in Los Alamos SOPs for Group WX-12 (SOP 27.5.1) and have been extended to all Los Alamos explosives areas.

III. DESCRIPTION OF THE SPOT-TEST KIT

The kit, illustrated in Fig. 1, consists of a commercially available deep-drawn seamless metal case 8.5 in. wide by 12 in. long by 6 in. high. The lid is 2.5 in. high to give a closed height of 8.5 in. The case looks similar to a train-travel cosmetics case. This configuration is stable when set on a truck bed because of its low center of gravity. A gasket seals the lid from the base, and the handle is on top of the lid so the reagent bottles are upright at all times. If the kit is upset, there is no spillage because the bottles are secured.

The inside of the case has been partitioned to hold the reagent bottles, the portable UV lamp (UVP Model G-14),* and charger, and the utensils and supplies needed for testing. The four reagent bottles are cushioned in a mold made of silicone rubber resistant to chemicals. Three test tubes are also cushioned to hold the dropper and stoppers to prevent contamination. Supplies include glass droppers, a spatula, filter papers, polyethylene bags, paper towels, a grease pencil, and instructions. The plastic bags are used to

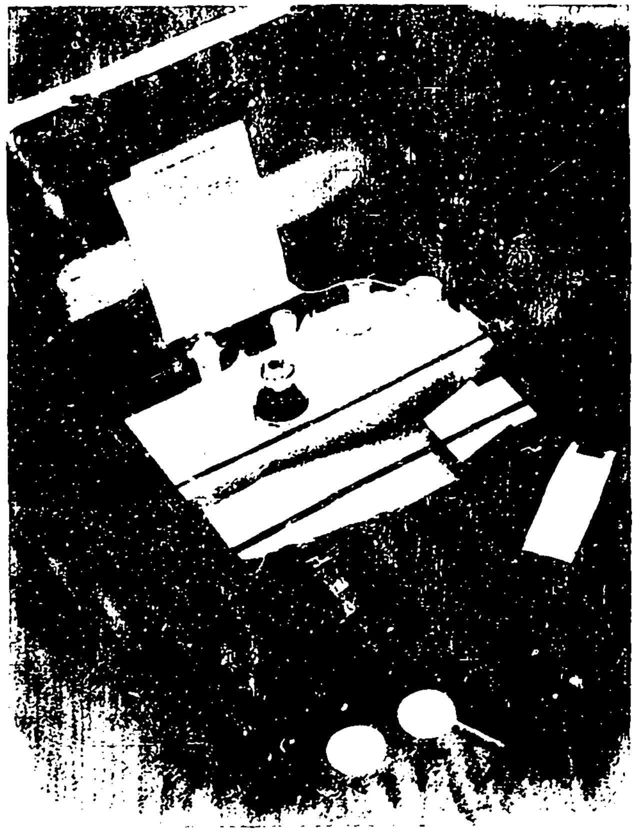

Fig. 1. Spot-test kit with reagents and filter papers. Shown also is a portable UV lamp. A test for TNT and a blank with Reagent A show blue-black color on the left circle and a clear blank on the right.

secure and transport samples to the analytical laboratory and to hold used test filters for disposal.

The reagents are in 50-ml glass-stoppered bottles. When the lid is closed, a semirigid foam cushion in the lid presses on the stoppers to prevent spillage. Reagents A, B, and C are described in the appendix; the fourth bottle contains acetone to clean spots with the paper towels.

This particular kit is the third revision and was designed for transportation stability and ease of use based on our experience with the previous models. No problems have been reported in two years of use. When the kit is packed and closed, the reagent bottles are secured so that any jostling and bouncing will not spill the contents. When the latches are opened and the lid is turned up, the kit is ready for use. In Fig. 1.

*Mention of commercial products and trademarks does not mean that Los Alamos endorses the product.

a filter paper with a swipe sample has been tested with Reagent A and shows a deep blue-black color, indicating explosives present. The control blank circle on the right is clear, showing the absence of explosives. This is evidence of the simplicity of operation.

ACKNOWLEDGMENTS

This will acknowledge the input of Bill McCormick, a constant user of this kit, for redesigning the kit to be more stable with a low center of gravity to withstand the rough and tumble use in transporting it in a truck to the field. I wish to thank B. W. Harris for the use of the spot test specific for TATB and for permitting its inclusion in this kit. The success of this spot-test kit depends on the use of the kit by technicians who have been trained in its use to allow the Laboratory to conduct its operations in a prescribed manner with safety as outlined in the SOPs.

REFERENCES

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4. B. W. Harris, "TATB, Strong Basic Reactions Provide Soluble Derivatives For a Simple Qualitative High Explosive Spot Test," Journal of Energetic Materials, Vol. 3, pp. 81–83 (1985).
5. B. W. Harris, "Spot Test for 1,3,5-Triamino-2,4,6-Trinitrobenzene, TATB," US Patent No. 4618452 (October, 1986).

APPENDIX

Detection of Explosive Materials in the Field

Reagent		Composition of solution
A	Develops dark blue-black color with cast (TNT) explosives immediately.	80%/20% by volume of N,N-dimethylformamide/di-n-butylamine.
B	Develops blue-black color under UV light in five minutes or less with plastic-bonded explosives (RDX, HMX).	50 mg dichlorofluorescein, 2 ml tetra-n-butylammonium hydroxide (10% solution in methanol), and 98 ml DMSO.
C	Develops red color for TATB, dark red under UV with TATB PBXs.	5 g KOH, 5 ml water, and 90 ml DMSO. Shake until solution is homogeneous.

Directions

1. Rub off some sample onto clean #2 Whatman filter paper. Dust off excess. Use one circle for each of three reagents.
2. Place on clean paper towel surface.
3. Add 1 drop of Reagent A to filter. Reagent will migrate and will give a black-to-purple color immediately with presence of TNT.
4. Add 1 drop of Reagent B to second filter paper sample. Reagent will migrate and react with RDX/HMX to give a blue-black color. Shine UV light on test area and it will give a black color, indicating RDX/HMX.
5. Add 1 drop of Reagent C to third filter paper sample if TATB is suspected. A red color will appear and will be enhanced with UV light.
6. Run a blank filter with reagents as a control.

General Information

1. Reagent A may be used confidently to −70°C, at which temperature it will form a slush.
2. Reagents are available at the Group M-1 Analytical Laboratory.
3. Apply a thin layer of silicone grease on ground stopper to prevent dripping.