

W. Fisco,¹ Ph.D.

A Portable Explosives Identification Kit for Field Use

Throughout the years, field analysis of explosives has often been a subject of interest and previous work at Picatinny Arsenal has led to the development of field kits for the qualitative analysis of explosives. Because the methods described in reports [1,2] utilize wet chemical reactions, they are not readily portable because of the bulky nature of the reagents involved. Furthermore, interpretation of the color reactions involved requires, in the least ambiguous cases, a degree of technical competence not readily acquired by personnel conducting field analyses. When sample specimens are limited in quantity, the application of a destructive method of identification may preclude the conducting of required specific identifications. Although Jenkins and Yallop [3] and Hansson [4] used conventional thin-layer chromatography (TLC) to generate useful chromatographic information, the method was, nevertheless, destructive by the application of visualization sprays resulting in color reactions.

In this work the approach was to develop a field kit that incorporated a nondestructive uniform analytical technique with emphasis on portability, ease of operation, and low cost. The report details the modification of a commercially available portable kit for the achievement of these objectives.

Experimental

TLC Plate

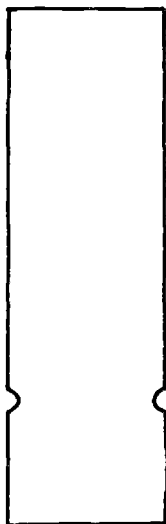
Polyethylene terephthalate sheets, precoated with silica gel containing a fluorescent indicator (N-HR/UV 254) supplied by Brinkman Instruments, Inc., Westbury, N.Y. were cut to 20 by 65-mm size. The plates were then notched as indicated in Fig. 1, for irrigation and spotting. This plate design represents a modification of the one supplied with the Kodak Chromat/O/Screen[®] kit (60-test kit) by the elimination of a center hole between the notches. The plates were cut carefully from the sheets to fit properly in the developing chamber. The plates were activated in a drying oven at 100°C for thirty minutes and stored in a desiccator or airtight container ready for use.

Developing Chamber

The chamber is supplied with the Kodak kit and is designed with a cover seal for the chromatographic plates in contact with the developing gel agent.

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¹ Research chemist, Chemistry Branch, Explosives Division, Feltman Research Laboratory, Picatinny Arsenal, Dover, N.J. 07801.



Notches made with a Keysort Punch to mark the point of sample introduction and regulate flow of developer.

FIG. 1—Actual size of chromatographic sheet.

Gel Developer

Cab-O-Sil[®], a thixotropic powder supplied by Packard Instrument Company, Inc., Downers Grove, Ill., was mixed separately with each of the following: (1) benzene, (2) 1:1 acetone:chloroform, (3) 5:5:4:1:1 toluene:benzene:*n*-hexane:*n*-pentane:acetone, (4) 1:1 benzene:nitromethane, and (5) 3:1 benzene:nitromethane. Enough developer was added to the Cab-O-Sil[®] to produce a gel with the property of retaining the form in the developing chamber resulting from the leveling action of the scraper. The gels were leveled with the scraper supplied in the Kodak kit.

Visualizer

The thin-layer chromatograms were visualized with a Raytech Model LS-88 ultraviolet (UV) lamp at 254 nm. The lamp can be used either with service line at 115V—60 cycles, or with a power pack containing two 45-V "B" batteries, supplied by Raytech Industries, Inc., Stafford Springs, Conn. Resolved constituents on the fluorescent silica gel appear as dark areas against a gray-green background. This unit is shown in Fig. 2.

Identification Procedure

Standards consisting of 1% acetone solutions of twelve military explosives were prepared in the laboratory. In an actual explosion investigation, dilute acetone extracts should be prepared at the postexplosion site of suspect fragments, in the vicinity of "ground zero," detected (1) with the unaided eye via visual evidence of explosives residues in the form of crystals or of blackened areas and (2) with a magnifying glass or microscope as trace surface contaminants.

The developing chamber was prepared with the appropriate gel developer. With a 1- μ l pipet supplied with the Kodak kit, one to two drops of the acetone solution were applied to the activated TLC plate at the notched sector and placed in the developing chamber in contact with the gel developer. Normal ascending chromatography was then allowed to

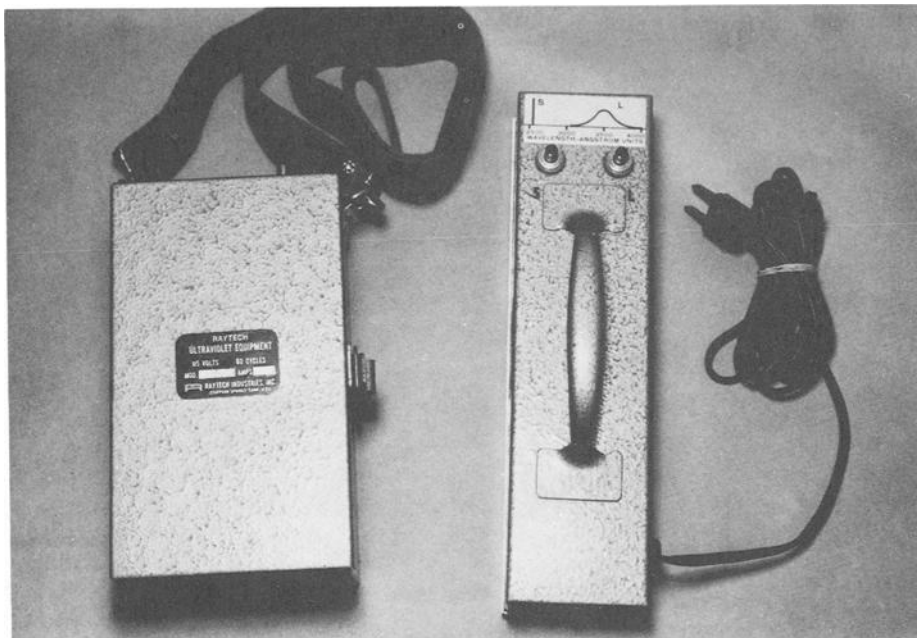


FIG. 2—The UV lamp and accompanying battery pack were found satisfactory for “visualizing” compounds on the chromatographic sheets.

proceed. After the usual 20 to 30 minutes for development, the TLC plate was removed and air-dried. Forced air may be used if available.

The R_f values of the resolved constituents were determined with the UV visualizer and compared with the standard reference values for identification. In an actual test case, if a given spot does not ascend beyond the notched sector, various gel developers should be used alternately in an attempt to obtain an R_f value.

Finally, TLC records should be subjected to high resolution instrumental methods of analyses for specific identification. The resolved constituents can be removed mechanically and introduced directly into the electron spectroscopy for chemical analysis (ESCA) or a mass spectrometer equipped with a solid probe to analyze solids.

Field Kit Components

The component parts of the field test kit for explosives are shown in Fig. 3 and a list of explosives reference standards with their respective R_f values for the various gel developers is given in Table 1. The UV lamp, operated in the battery mode for field use, is described in the above Visualizer section. A magnifying glass or microscope (not shown) or both should be integral parts of the field test kit.

Recommended stock items are listed as follows:

- (1) precoated plastic sheets;
- (2) chemical solvents, preferably spectral grade: acetone, benzene, chloroform, toluene, *n*-hexane, *n*-pentane, and nitromethane;
- (3) Cab-O-Sil®;



FIG. 3—The TLC items pictured are, from left to right, plastic developing chamber, prepared gel stored in screw-top test tube, acetone, 1- μ l pipets and holder, instruction booklet, scraper, chromatographic sheet, and plastic forceps. Standard items such as spatulas, labels, vials, and tissue wipes are not shown.

- (4) stoppered vials and labels; and
- (5) cotton swabs, spatulas, tweezers, and probes.

Results and Discussion

At the onset of this investigation it was anticipated that the Kodak kits could be used as received for the identification of explosives in the field. The Chromo/O/Screen[®] kit for alkaloids was selected and purchased for this purpose. Attempts to resolve a mixture of 2,4,6-trinitrotoluene (TNT), 1,3,5-trinitrobenzene (TNB), and 2,4,6-trinitrophenyl *N*-methylnitramine (tetryl), using the gel developer supplied with the kit, resulted in the migration of all three constituents at the same velocity as the solvent front. Some introductory textbooks on TLC [5,6] and a review of current literature [7] are available.

The gel developer supplied with the Kodak kit contains ethyl acetate, methanol, ammonium hydroxide, and gelling agent (fumed silica) [8]. To effect a resolution of the mixture of these nitroaromatics by the promotion of differential sorption on the silica gel, the polarity of the gel developer supplied with the kit was changed. A new gel was prepared consisting of benzene/Cab-O-Sil[®] which proved to be satisfactory. The resolved explosives were visualized in the conventional manner by spraying the TLC plate with a 10% morpholine in acetone solution which gave sharply colored spots.

Visualization of explosives and related compounds on TLC plates by the color reaction method has been reported by other investigators [3,4,7-17]. Color reactions have also been

TABLE 1— R_f values of twelve explosives for various developers.

Compound	R_f Values			
	Benzene	1:1 Acetone: Chloroform	<i>n</i> -hexane: <i>n</i> -pentane: Acetone	1:1 Benzene: Nitromethane
1. Trinitrotoluene, 2,4,6	0.68	...	0.70	...
2. Trinitrobenzene, 1,3,5	0.58	...	0.66	...
3. 2,4,6 trinitrophenylmethyl- nitramine (tetryl)	0.40	...	0.44	...
4. Hexa-hydro-1,3,5 trinitro-s- triazine (RDX)	0.10	...	0.12	0.80
5. Octo-hydro-1,3,5,7 tetranitro-s- triazine (HMX)	0.00	0.70
6. Nitroglycerin ^a	0.66	0.86
7. Pentaerythritol tetranitrate (PETN) ^b	0.00	0.92
8. Nitroguanidine	0.00	0.24
9. Picramide	0.36
10. Picric Acid ^c	0.34
11. Ammonium picrate (Explosive D)	0.00
12. Ethylenedinitramine (haleite)	0.00

^a 10⁻⁵ g were used.^b 5 × 10⁴ g/spot were used.^c Streaks the chromatogram, R_f calculated on maximum distance travelled.

used for the quantitative determination of explosives [18]. However, when hexa-hydro-1,3,5-trinitro-s-triazine (RDX) was tried, no color reaction was observed. At this point in the investigation it became apparent that visualization of nonaromatic explosives on TLC plates would become increasingly difficult if color reactions were to be used in the identification scheme. Chemical color reactions do exist for RDX as well as for other nitramine explosives, and have been done on specially prepared TLC plates [19]. This, however, represents a tedious task to even experienced workers and is not desirable in a field kit.

TLC separations of explosives have been reported using silica gel containing some fluorescent indicator [20,21]. The compounds appear as a dark spot against the gray-green fluorescent background when illuminated with UV light at 254 nm. This type of visualization eliminates the need for wet chemical color reactions if the compound is a good absorber of UV light, as many explosives are, and adds a dimension of flexibility to the field testing kit. The fluorescent indicator used is manganese-activated zinc silicate, which has a maximum absorption at 254 nm [22]. Whereas the color reaction method is limited and destructive in its application for the identification of explosives on TLC plates, the UV visualization technique is more inclusive and nondestructive. However, it should be noted that if an explosive is neither soluble in the acetone extract or a nonabsorber of UV light, the kit would not be amenable for these analyses.

After preliminary identification with the UV lamp the explosive is in a pure and resolved state, in a form ready for subsequent confirmatory analysis. For the more volatile explosives, for example, nitroglycerin (NG), its isolation in the adsorbed state on silica gel prolongs residence time for subsequent analyses. Although the explosive exists in microgram quantities, with the availability of high resolution and high sensitivity instrumentation in most laboratories today, its unambiguous identification can be considered routine. The use of ESCA has proven to be an effective tool for confirmatory analysis of trace quantities of explosives present in surface layers. Sample specimens scraped from the TLC plate have been analyzed in the case of NG isolated from the scene of an explosion. The concept of identifying TLC records directly is also applicable to mass spectrometric analyzers equipped with a solid probe for the introduction of the explosive adsorbed on the silica gel.

The R_f values for the twelve military explosives listed in Table 1 were obtained by the application of the UV visualization technique. The samples were either composite explosives (for example, tetrytol) or neat explosives of military grade. In most cases microgram quantities of sample were spotted on the TLC plate; variations of this amount are noted in Table 1. Advantages have been reported [23,24] in the use of TLC chromatographic plates the size of a microscope slide. Because the chromatographic plates used in this study are approximately of this size, further credence may be given to using R_f values as an aid in the tentative identification of explosive compounds. For any given explosive listed, the R_f value, generated by the specified developer, can serve as a reference standard in the process of making a preliminary identification in the field.

The explosives field test kit has been designed for its ease of employment, which does not require the user to have any background in chemistry. One requirement placed on the user is that he obtains postexplosion sample specimens for TLC examination with discrimination. This requirement should apply in general to any method used for the postexplosion identification of explosives. The usual practice of collecting massive amounts of debris and fragments for extraction in the laboratory precludes the possibility of identifying trace quantities, because of the large interfering background incorporated in the final concentrated extract. Instead, by the careful removal of what appears to be explosive fragments, the user of the kit can effectively screen the area to isolate explosives specimens for substantive identification in the laboratory. The role played by the user of the kit in the field

is therefore to isolate the potential explosive fragment with the aid of tweezers, magnifying glass, and microscope if available and transfer it to the TLC plate, where it is resolved and maintained in a quasi-permanent form. Once in this form the sample specimen, though in trace quantities, is ready for precise analysis in the laboratory by qualified scientists trained in the use of sensitive instrumental methods of identification. Oftentimes when trace quantities of volatile explosives are not entrained or fixed in any way at the postexplosion scene, the specimen is lost before it reaches the laboratory.

During the process of concentrating extracts from postexplosion debris and fragments submitted to the laboratory, microgram quantities of the more volatile explosives may in some instances be lost. Furthermore, even when isolated, whether volatile or not, such trace quantities are sometimes insufficient for the more conventional methods of analyses such as infrared spectrophotometry, nuclear magnetic resonance spectrometry, and wet chemical methods. In such cases, application of the miniaturized TLC technique in the laboratory to dilute extract solutions has been found to be effective. TLC chromatograms of the resolved and fixed explosive specimens in microgram quantities have been identified by the trace methods of analysis cited above. This method appears to offer definite advantages over some of the more classical identification techniques such as the diphenylamine test.

Summary

This study has shown the feasibility of using a miniaturized TLC kit for the presumptive identification of explosives in the field. The process of TLC field screening of a post-explosion site is nondestructive, resolves constituent explosives from interfering backgrounds, and increases the residence time of isolated specimens in the adsorbed state for subsequent substantive identification by trace methods of instrumental analyses.

The cost of the modified Kodak kit for explosives identification (excluding the microscope) is approximately \$150. The stock items, such as the fluorescent silica gel sheets, Cab-O-Sil[®], and solvents, are of nominal cost. The kit is relatively simple to use and personnel with a minimal technical background can be trained in its use.

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Chemistry Branch, Explosives Division
Feltman Research Laboratory
Picatinny Arsenal
Dover, N.J. 07801