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Analysis of Explosives and Explosive Residues. Part 2: Thin-Layer Chromatography

In Part 1 of this paper [1] the chemical tests used by this laboratory in the screening of explosives and explosive residues were discussed. Part 2 presents the thin-layer chromatographic (TLC) methods used in this laboratory for the confirmation of commonly used organic explosive compounds.

Jenkins and Yallop [2] describe their TLC techniques for the determination of certain organic explosives. They used three different solvent systems (benzene, ethanol, and 1:1 chloroform-acetone) and migrated the solvent front for 19.25 cm. Unfortunately, they do not study each compound in all solvent systems used. Hoffman and Byall [3] also present R_f values for EGDN and NG in various solvent systems (see Table 1 for abbreviations of compounds).

TABLE 1—*Abbreviations of organic explosives studied.*

2,4-DNT	2,4-dinitrotoluene
2,6-DNT	2,6-dinitrotoluene
EGDN	ethyleneglycol dinitrate
NC	nitrocellulose
NG	nitroglycerin
NS	nitrostarch
RDX	cyclotrimethylenetrinitramine
PETN	pentaerythryl tetranitrate
Tetryl	2,4,6-trinitrophenylmethylnitramine
TNT	2,4,6-trinitrotoluene

Procedure

In Part 1 the examination and extraction of explosives and explosive residues was discussed. The acetone extract from these procedures is used for TLC analysis. Glass plates, 8 by 2 in., precoated with a 250- μ m thickness of Silica Gel G (Analtech, Inc.) are used. The concentrated acetone extract is spotted on the plate along with a reference standard. The plate is developed for 10 cm with chloroform in a 2¼-in. cylindrical tank. The plate is removed and eluent evaporated. The plate is then taken through a three-stage process to develop colors. In Stage 1 the plate is placed in a spray box inside a

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fume hood and sprayed with 5% diphenylamine in 95% ethanol. Any colors and R_f values are noted. In Stage 2 the plate is placed under ultraviolet (UV) light (Chromato-Vue®, Ultra-Violet Products, Inc.) for 5 to 15 min. Again colors and R_f values are noted. In Stage 3 the plate is again placed in the spray box and sprayed with concentrated sulfuric acid. The results are compared with the standard on the plate and knowns given in Table 2.

TABLE 2—Color development of TLC spots.

Compound	Color Development			R_f in Chloroform
	Stage 1, Diphenylamine	Stage 2, UV Light	Stage 3, Sulfuric Acid	
2,4-DNT	yellow	yellow	color fades ^a	0.8
2,6-DNT	yellow	yellow	color fades ^{a, b}	0.85
EGDN	NCD	gray to gray-green	blue-gray	0.8
NC	NCD	gray to gray-green	blue to blue-gray	0
NG	NCD	gray to gray-green	blue-gray	0.7
NS	NCD	gray to gray-green	blue to blue-gray	0
RDX	NCD	gray	blue-gray	0.15
PETN	NCD	gray to gray-green	blue to gray-green	0.7
Tetryl	brown	brown	yellow to blue-gray ^c	0.5
TNT	orange-brown	orange-brown	color fades ^a	0.75

NCD = no color development

^a Dark spot visible under UV light.

^b Turns pink in center after reexposure to UV light.

^c Yellow in center with blue-gray edge.

Results and Discussion

Table 2 gives approximate R_f values in chloroform and the colors developed in each stage for various organic explosives. While the nitrotoluenes have R_f values similar to EGDN and NG, they are easily distinguished by their initial color development in Stage 1. Both RDX and PETN can easily overload the plate and quite often streak from the origin.

A blue to gray color at the origin is indicative of any combination of inorganic nitrites, nitrates, nitrocellulose, and nitrostarch. If necessary, an infrared (IR) spectrum can be run on this acetone extract to obtain further information and confirmation of the substances present.

Table 3 gives approximate R_f values for the various explosives eluted in chloroform and the solvent systems used by Hoffman and Byall [3] and Jenkins and Yallop [2]. In our laboratory we prefer to elute with chloroform, primarily because of the convenience of using a single solvent while giving adequate separations. In most cases the R_f values and color development are sufficient for confirmation. If necessary, a second solvent system may be used.

A solvent system giving satisfactory migrations for nitrostarch or nitrocellulose has not been found. Also, in none of the systems studied have PETN or NG given satisfactory separation. If there is only a single spot at the approximate R_f for NG or PETN and none for EGDN, then PETN should be suspected to be present since EGDN is almost always present with NG in dynamites. As mentioned previously, PETN easily streaks whereas we have never noticed NG or EGDN to give comparable streaking. This could

TABLE 3—TLC R_f values in various solvent systems.

Solvent System	Compound									
	2,4-DNT	2,6-DNT	EGDN	NC	NG	NS	RDX	PETN	Tetryl	TNT
Chloroform	0.8	0.85	0.8	0	0.7	0	0.15	0.7	0.5	0.75
CCl ₄ /Dichloroethane (4:1)	0.5	0.5	0.5	0	0.35	0	0.02	0.3	0.1	0.45
Benzene/Hexane (1:1)	0.5	0.55	0.5	0	0.35	0	0.02	0.3	0.1	0.45
Xylene/Hexane (3:2)	0.4	0.45	0.5	0	0.35	0	0.03	0.3	0.15	0.4
Benzene	0.7	0.75	0.75	0	0.65	0	0.1	0.65	0.45	0.75
Hexane/Acetone (4:1)	0.4 ^b	0.45	0.4	0	0.4	0	0.2	0.4	0.3	0.4
Chloroform/Acetone (1:1)	0.95	0.9	0.9	0	0.9	1.0 ^c	0.7	0.95	0.9	0.9
							0.47 ^b	0.69 ^b	0.66 ^b	

^a Hoffman and Byall [3].

^b Jenkins and Yallop [2].

^c Continuous streaking from origin.

be used as a distinguishing characteristic if a sufficient amount of PETN is present. PETN may also be eluted by an alternate solvent system such as benzene-hexane (1:1), which does give a slight separation between NG and PETN. It may also be confirmed by IR.

Unfortunately, the acetone extraction also carries with it many impurities from the sample extracted. These impurities generally appear in a chromatogram as a long streak which is highly fluorescent under UV and which may show multiple colors when sprayed with reagents. This may decrease the sensitivity of TLC to explosives. As mentioned in the previous paper, one partial solution to this problem is to completely evaporate the acetone and to take up the extraction in methanol or ether. Quite often this will considerably reduce the impurities showing up in TLC. It has the disadvantage that many explosives are less soluble in methanol and ether than in acetone.

From the results given here, it is apparent that TLC is the method of choice to confirm the presence of the organic explosives studied. Thus, this method is well suited to the analysis of bomb scene residues.

References

- [1] Parker, R. G., Stephenson, M. O., McOwen, J. M., Cherolis, J. A., "Analysis of Explosives and Explosive Residues. Part 1: Chemical Tests," *Journal of Forensic Sciences*, JFSCA, Vol. 20, No. 1, Jan. 1975, pp. 133-140.
- [2] Jenkins, R. and Yallop, J. "The Identification of Explosives in Trace Quantities on Objects Near an Explosion," *Explosivstoffe*, Vol. 6, 1970, pp. 139-141.
- [3] Hoffman, C. M. and Byall, E. B., "Identification of Explosive Residues in Bomb Scene Investigations," *Journal of Forensic Sciences*, JFSCA, Vol. 19, No. 1, Jan. 1974, pp. 54-63.

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