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Joseph Almog^a; Shmuel Kraus^a; Baruch Glattstein^a

^a Division of Criminal Identification, Israel National Police, Jerusalem, Israel

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ETK-AN OPERATIONAL EXPLOSIVE TESTING KIT

Joseph Almog,* Shmuel Kraus, and Baruch Glattstein

Division of Criminal Identification,
Israel National Police,
Jerusalem, Israel

ABSTRACT

A chemical kit for field determination of traces of explosives on hands of suspects is described. The system involves three components: dry sampling devices ("detection buttons"), and two tubes of chemical reagents. It detects virtually all the common military explosives, and there is also an option to detect home-made explosives based on inorganic fertilizers. Detection is based on two well known chemical reactions for which optimal conditions have been developed. In stage I polynitro aromatics are detected by the formation of colored Meisenheimer complexes. In stage II organic nitrates and nitramines are consequently detected by modified Griess test.

* Author to whom correspondence should be addressed.

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THE PROBLEM

The past several years have seen an alarming increase in the use of explosives in criminal and terrorist activities. In such circumstances many suspects must be screened quickly for recent contact with explosives; and police, customs and other security agencies must be able to determine rapidly whether a suspected substance is an explosive. The requirement presented to us in the late 1970's was for a simple, quick, reliable and inexpensive device for the detection of traces of explosives on hands of suspects and on objects, and the ability to distinguish between explosive and non-explosive material.

WHY A CHEMICAL KIT?

Explosive-vapor detectors are relatively complicated and expensive. Dogs require an elaborate and expensive logistical system of training and are not always reliable. Thus, we decided to study the possibility of utilizing "wet chemistry".

The idea was to find the shortest sequence of chemical spot-tests which would indicate the presence of minute amounts of explosives by formation of specific colors. Indeed, we found a very short scheme that, by a minimal number of reactions, enables the detection of practically all military explosives. By modifying the chemical composition of some known reagents, we managed to shorten the reaction time and not less important-to carry out the entire process at room temperature. The assembly received the name Explosive Testing Kit (ETK).

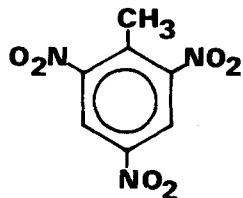
CHEMISTRY OF ETK

The vast majority of military explosives contain one of the three following chemical groups: polynitro aromatics, organic nitrates (nitrate esters) and nitramines (Figure 1).

A thorough literature survey¹ as well as numerous laboratory experiments led us to the conclusion that the shortest and most economical sequence for the formation of colored products from all the above military explosives is the following (Figure 2):

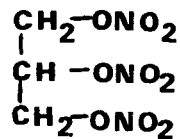
Polynitro-aromatics, such as TNT, TNB, picric acid and its derivatives, form highly colored compounds (Meisenheimer complexes) upon reaction with alkali. Common alkali² formulations for explosives analysis contain 5-10% KOH or NaOH in ethanol or water³. Nitrate esters such as NG, EGDN, PETN and NC under similar conditions, undergo alkaline hydrolysis⁴ producing nitrite ions (NO_2^-) which can be readily detected by the well known Griess reaction⁵⁻⁷. This includes the action of NO_2^- ions on an aromatic amine, usually sulfanilic acid, in an acidic medium. The diazonium ion thus formed is then coupled with an aromatic nucleophile (usually α -naphthyl amine) to produce a colored azo dye. Nitramines such as RDX, HMX and tetryl too, undergo alkaline cleavage to form nitrite ions, NO_2^- , which produce the same colored azo compound by the Griess reaction^{8,9}. When the above scheme (Figure 2) was applied to actual explosives, it was found that while polynitro-aromatic explosives reacted as expected,

1. Poly-nitro Aromatics. Example: **TNT**



2. Organic Nitrates. Example:

NG



3. Nitramines. Example:

RDX

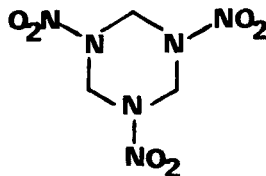


FIGURE 1

Most common chemical groups of military explosives.

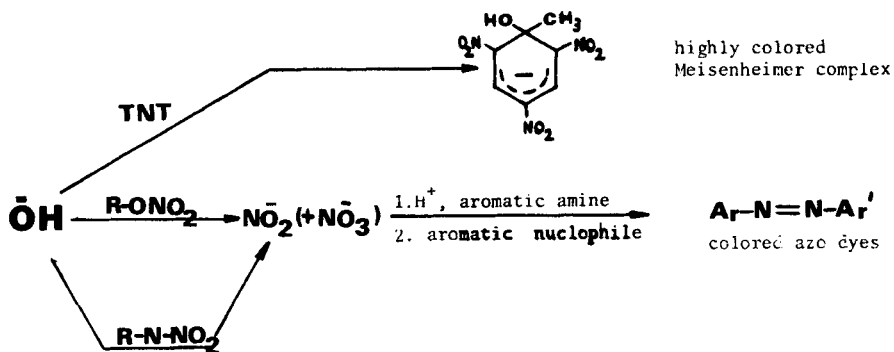


FIGURE 2

Formation of colored products by the reaction of military explosives with alkali.

certain plastic explosives (and particularly those which are based on nitrate esters) reacted very slowly and required heat for the color formation. This, of course, was a great disadvantage for field work.

An improvement was achieved when dimethyl sulfoxide (DMSO) was used as the major solvent for the alkaline reagent instead of the alcoholic or aqueous solutions that were previously used. This change accelerated the color formation with plastic explosives such as Detasheet (mostly PETN + binder) or Semtex - H (RDX+PETN+Binder).

A second improvement was achieved by the addition of reducing agents such as mercaptanes, ascorbic acid or hydrazine to the alkaline solution. This was effective with explosives containing nitrate esters.

The optimal composition for stage 1 (alkaline reagent) was found to include KOH in EtOH/DMSO with a small amount of reducing agent. A plausible explanation for the advantages of this formulation over the alcoholic-KOH reagent might be the following:

- a. DMSO is a better solvent for plastic explosives (particularly for the binder), than water or alcohol.

b. Nitrate esters, under alkaline conditions, produce not only nitrite ions (NO_2^-) but also nitrate ions (NO_3^-)¹⁰ which reduce the detectability of the parent compound (as the NO_3^- ions do not participate in the Griess reaction). The reducing agent in the above composition converts the nitrate ions (NO_3^-) to nitrite ions (NO_2^-) which increases the sensitivity of the detection (reduction of nitrate to nitrite by hydrazine in alkaline solution was studied in detail by Sawicki and Scaringelli¹¹).

A combination of sulfanilamide and N-(1-naphthyl)ethylenediamine for the diazotation and coupling-stage¹¹ gave more satisfactory results than the more common formulation of sulfanilic acid and α -naphthylamine. The reaction was faster and the color intensity-higher (Also, the formerly used α -naphthyl amine is known to be carcinogenic).

SAMPLING METHOD

From the very beginning of this project we decided to adopt a "dry sampling" method, the rationale being that if a positive indication for the presence of explosive is obtained, there is still a sufficient amount of material left on the object for laboratory confirmation (The efficiency of "wet sampling" methods such as cotton-wool swabbing or hand-washing with solvents is much higher, thus leaving very little on the object for further examination).

The sampling devices that were designed for this purpose are "detection buttons" (Figure 3). They are composed of a circle of the coarse filter paper, compressed between a plastic disc and a plastic ring. They enable the technician to apply pressure against the fingertips of suspects without contaminating the sampling surface. For sampling surfaces other than fingers, square or round pieces of the same filter paper are used.

SENSITIVITY

The limits of detection by ETK were determined independently by the Metropolitan Police Laboratories in London and by this group. The human eye was used as detector. It was found to give a visible reaction with amounts as small as 10^{-7} g TNT per button and 10^{-8} g NG.

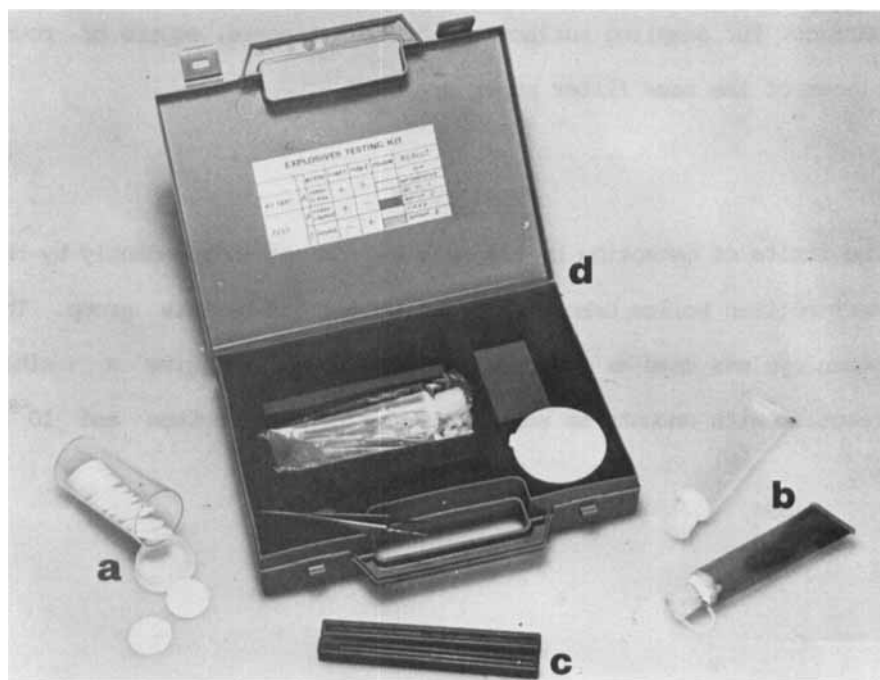


FIGURE 3

- Components of ETK
- a. Detection buttons
 - b. Reagent tubes.
 - c. Ampule cracker
 - d. Box

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