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*An Attempt to Identify an Explosive
in a Highly Confined Detonation*

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3 9338 00313 9952

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This work was supported by the US Air Force Technical Applications Command.

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LA-8965-MS

UC-45

Issued: September 1981

An Attempt to Identify an Explosive after Highly Confined Detonation



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AN ATTEMPT TO IDENTIFY AN EXPLOSIVE AFTER
HIGHLY CONFINED DETONATION

by

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ABSTRACT

Experiments have been performed to determine whether an explosive composition can be ascertained from the direct sampling and analysis of the gases and residues left after a highly confined detonation in air. The explosives used in this study were PBX 9501, PBX 9502, and TNT, which were doped with fluorescein dye. Charges were fired unconfined, partially confined, and fully confined in lead and in stainless steel. Residues were taken from particle filters and from a polyethylene sheet placed in the vicinity of the detonation. Residues were analyzed by Electron Spectroscopy for Chemical Analysis (ESCA) and spectrophotometrically. Gases were analyzed by mass spectrometry. The results of this study show that no gases or residues that are indicative of the explosive detonated survive the explosion.

I. INTRODUCTION AND BACKGROUND

This work resulted from an October 1979 meeting between R. J. Morrow (Air Force Technical Applications Center Nuclear Division), D. R. Worlton (Los Alamos National Laboratory Nuclear Applications Group X-4), and several members of the Explosives Technology Group (WX-2). The purpose of this meeting was to discuss what identifiable explosive residues, if any, would survive the detonation of a well-designed implosion system, and how such residues could be collected and analyzed to give evidence of what explosive was used. The consensus from the WX-2 explosive experts was that no identifiable residues or gases would remain after such a detonation.

R. J. Morrow pointed out that he had heard such opinions before, but that he was unaware of any specific experimental evidence. Therefore, WX-2 offered to do experiments on high explosives of

widely different oxygen balance and attempt to identify residues and gases that might be used as a signature of the explosive used.

The experiment proposed by WX-2 was a simple one, involving the direct, intimate sampling of a highly confined detonation. It was felt that if any signature substance did survive the explosion it must be sampled during the event or within a few seconds afterward.

II. EXPERIMENTAL METHOD

Figure 1 shows the explosive charge configuration used in this study. The explosives fired were 10-g charges of TNT and of PBX 9501,* and 5 g of PBX 9502** boosted by 5 g of PBX 9501.

The first experiments used several layers of 3.2-mm-thick lead foil for partial confinement (the ends were left open). Later experiments used 25.4-mm-o.d. stainless steel pipe with stainless steel threaded caps for complete confinement.

Figure 2 details the initial experimental design and sampling system. The charge was suspended inside a 25.4-mm-thick steel cylinder to contain fragments. The outer cylinder supported a steel plate on which were mounted the breakseals and particle filters. The outer cylinder also served to contain explosion products, allowing a brief moment when products were somewhat concentrated.

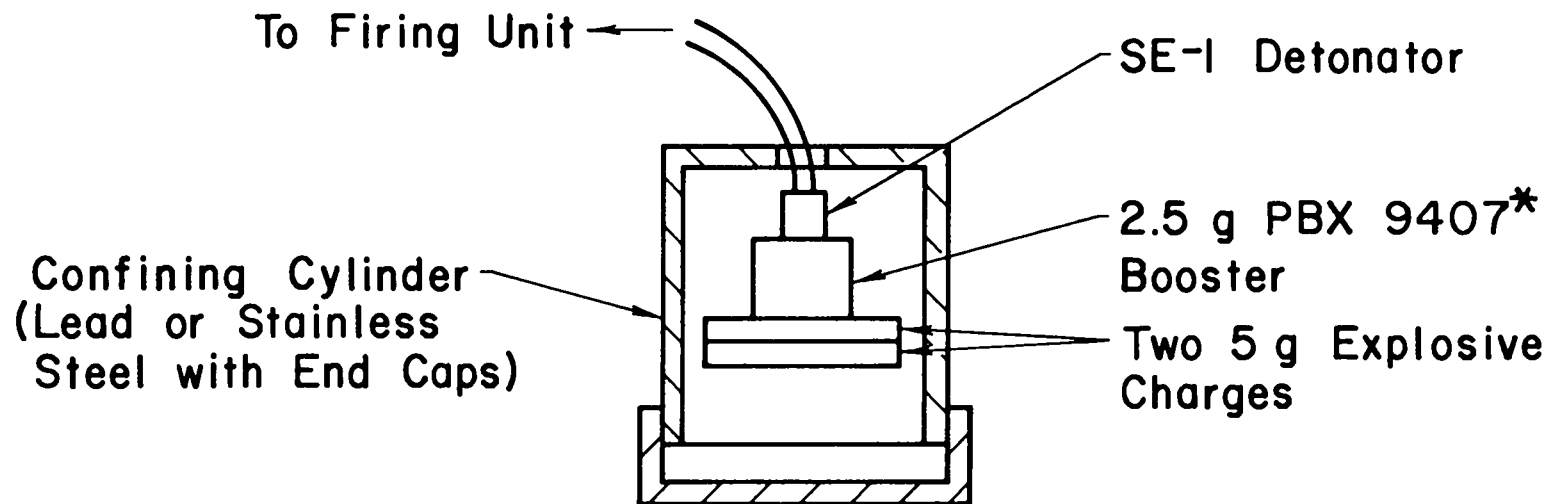
The force of the explosion was used to shatter glass-to-metal breakseals, allowing a gas sample to be drawn into an evacuated 24-L steel volume and allowing a volume of smoke and debris to be drawn through the particle filters by a 500-L/min vacuum pump. The exact conductance of the lines and filters was not measured, but typically the filters were pumped for 30 s.

The collected gas sample was bled from the 24-L volume through a fine needle valve into the sampling system of a UTI Model 100C Mass Analyzer (0-300 mass units). Mass spectra were recorded while a new test was being set up.

Residues, collected on the filters, were dissolved in acetonitrile (spectroscopic grade), and the solutions were analyzed spectrophotometrically (690-250 nm) on a Perkin-Elmer UV-Visible Spectrophotometer (Model 554). The filter pore sizes ranged from 0.25-15 μm , and the filters were high-conductance Millipore filters. The insolubles left on the filters were examined by Electron Spectroscopy for Chemical Analysis (ESCA) for any evidence of explosive residue.

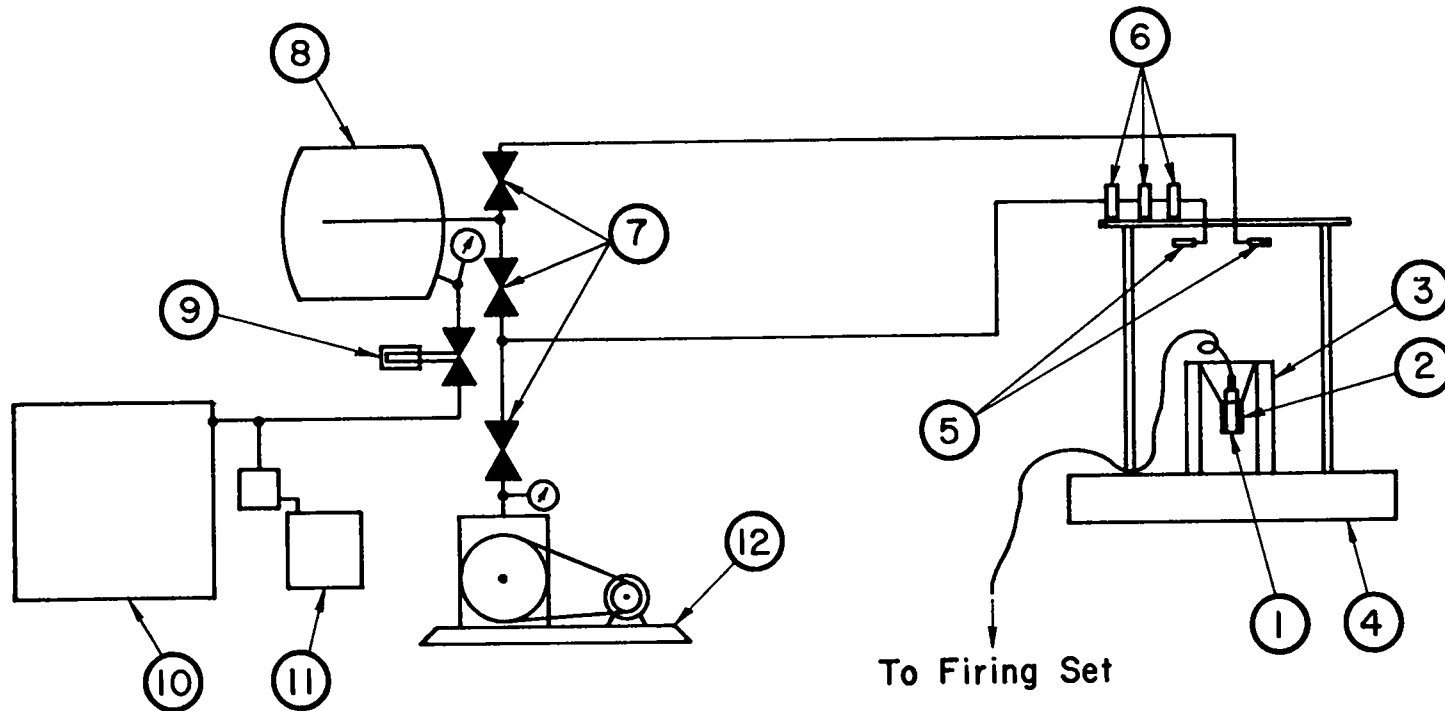
*PBX 9501 - 95/2.5/2.5 wt% HMX/Estane 5703/BDNPA-BDNPF eutectic

**PBX 9502 - 95/5 wt% TATB/Kel-F



* 95/5 wt% RDX/Exon 461

Fig. 1. Explosive charge configuration.



- | | | |
|-------------------|------------------------------|--------------------------------|
| 1) Explosive | 5) Glass-to-Metal Breakseals | 9) Needle Valve |
| 2) Containment | 6) Particle Filters | 10) Mass Analyzer |
| 3) Steel Cylinder | 7) Valves | 11) Baratron Pressure Gage |
| 4) Steel Block | 8) 24-L Steel Volume | 12) Vacuum Pump
(500 L/min) |

Fig. 2. Initial experimental setup.

Figure 3 illustrates a later experimental setup designed to improve sampling. The 9.1-m, 6.35-mm-o.d. stainless steel tubing and 24-L steel volume were replaced with a 0.914-m, 25.4-mm-o.d. section of copper pipe separated from a 10-L stainless steel volume by a remotely operated, pneumatic 25.4-mm ball valve. The end of the copper pipe was fitted with a 25.4-mm-o.d. glass-to-metal break-seal, which was positioned just a few centimeters from the explosive charge. In this way a gas sample was taken in 3 s, compared with the 15-20 s sampling time through the 12.7-mm-o.d. tubing.

After each experiment a 1-L aliquot gas sample was taken from the 10-L volume and mass spectrometrically analyzed on a high-resolution DuPont 21-104 Mass Spectrometer.

A 1.6-mm-thick sheet of polyethylene was wrapped around the inside of the inner steel cylinder to catch fragments, debris, and residues. After each experiment the plastic sheet was washed with solvent, and this solution was concentrated for spectrophotometric analysis. The sheet was then visually inspected for sooty areas, residues, and fragments. Selected areas were cut out and analyzed by ESCA.

Additionally, all explosives were tagged with 1.0 wt% of fluorescein dye. This dye is easily detected spectrophotometrically below 1 ppm. The assumption made here is that if the dye residue was not detectable then explosive residue was very unlikely to be present.

III. RESULTS AND DISCUSSION

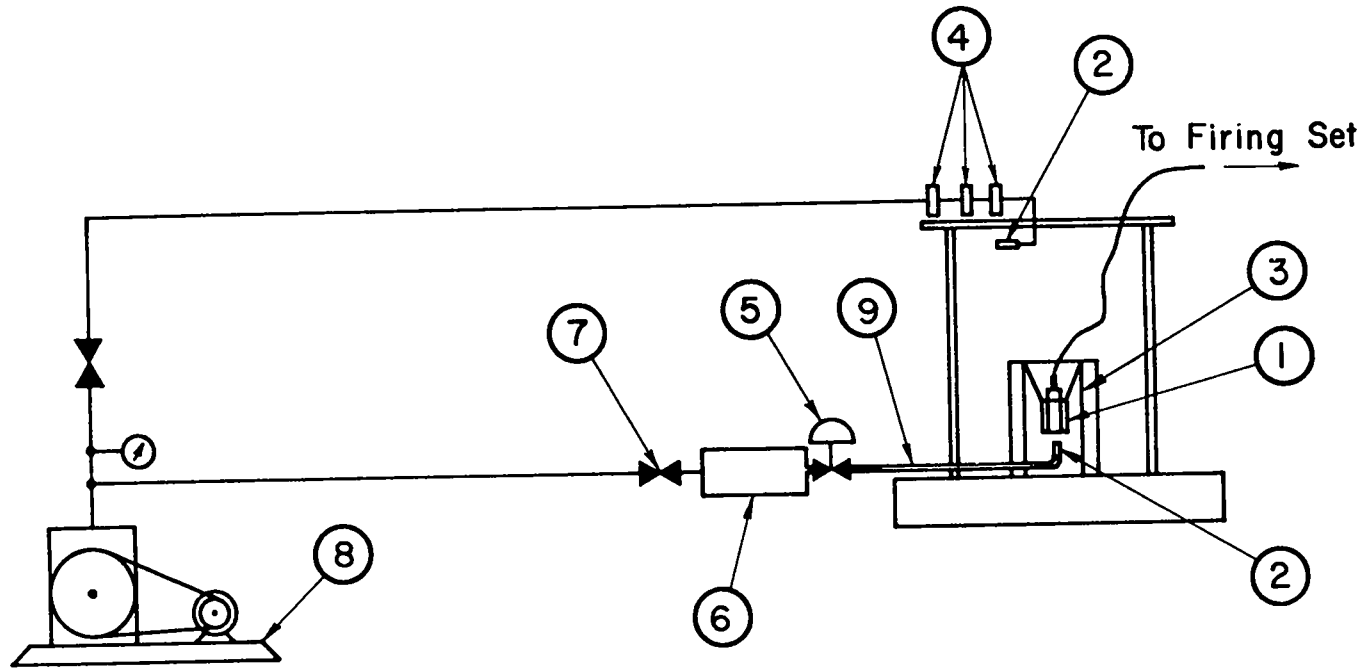
A. Initial Experiments

The first experiments (see Fig. 2) were done on 10-g charges of PBX 9501, PBX 9502, and TNT. These shots were fired unconfined and partially confined in lead foil. Lead proved to be a poor choice for confinement, because the particle filters quickly clogged with white lead oxide.

Residues from the filters were analyzed spectrophotometrically from acetonitrile solutions and compared with TNT, HMX, and TATB standard solutions. Insoluble residues were examined by ESCA for any trace of functional nitrogen, that is, as $-\text{NO}_2$, $-\text{NH}_2$, or any other chemical form.

No trace of functional nitrogen was found by any method, not even from unconfined shots.

The first 10-g charge of PBX 9502 fired did yield explosive residue, but the residue collected was obviously yellow, and it was thought that the insensitive explosive had not gone high order. A second experiment was done with 5 g of PBX 9502 boosted by 5 g of PBX 9501, and no residue was found.



- | | | |
|------------------------------|---------------------|-----------------------------|
| 1) Confined Charge | 4) Particle Filters | 7) Shutoff Valve |
| 2) Glass-to-Metal Breakseals | 5) Pneumatic Valve | 8) Vacuum Pump |
| 3) Polyethylene Lining | 6) 10-L Volume | 9) 25.4 mm-o.d. Copper Line |

Fig. 3. Final experimental setup.

In all cases, the gas analyses showed only slightly elevated CO₂ concentrations. It was very difficult to distinguish between the explosion samples and background air.

B. Final Experiments

The experimental apparatus was changed (see Fig. 3) to improve our sampling techniques. The explosives used were TNT, PBX 9501, and PBX 9501/PBX 9502 (5 g/5 g) in 10-g charges confined by stainless steel. Shots were fired unconfined, partially confined, and completely confined. A total of twenty shots were fired, of which about half were TNT, which we felt was most likely to leave a residue. All explosives were doped with 1.0 wt% fluorescein dye. Gas sampling times were reduced by a factor of 10.

The gas analyses were carried out on a high-resolution DuPont 21-104 Mass Spectrometer. In all cases, only increased CO₂ concentrations were detected. For example, the ratio of argon-to-CO₂ was 1.79, 2.54, and 2.47 for unconfined, partially confined, and fully confined TNT explosions. These ratios are compared to 0.03 for normal air. Similar numbers were found for the other explosives with no evident trend. One unconfined PBX 9501/PBX 9502 test showed traces of acetylene, but this result was not reproducible.

Figure 4 is an example of the spectrophotometric data from the methanol washings of the polyethylene sheet to detect fluorescein. The data of Fig. 4 are for PBX 9501/fluorescein unconfined and fully confined detonations. There is some evidence of fluorescein from the unconfined explosion, but no indication of dye survival from the confined shot.

Figure 5 shows similar data for unconfined TNT/fluorescein from the methanol extraction of the plastic sheet and the particle filters. In this case, there is no evidence of fluorescein survival in an unconfined shot.

Figure 6 is an example of the data obtained from ESCA scans of the plastic sheet from a fully confined PBX 9501 experiment. There is no evidence of functional nitrogen in any form. The scan shows carbon, oxygen, lead, silicon, sodium, and chlorine. The lead is debris from earlier lead-confined shots. Silicon dioxide and sodium chloride are common contaminants. Carbon was commonly collected as a residue in TNT and TATB explosions, but as it contained no functional nitrogen the carbon residue could not serve as an explosive signature.

IV. SUMMARY AND CONCLUSION

Experiments have been done, using intimate sampling techniques, to determine whether, in the detonation products from highly confined explosives, there existed a substance, or combination of substances, that could serve as a signature of the explosive used. No

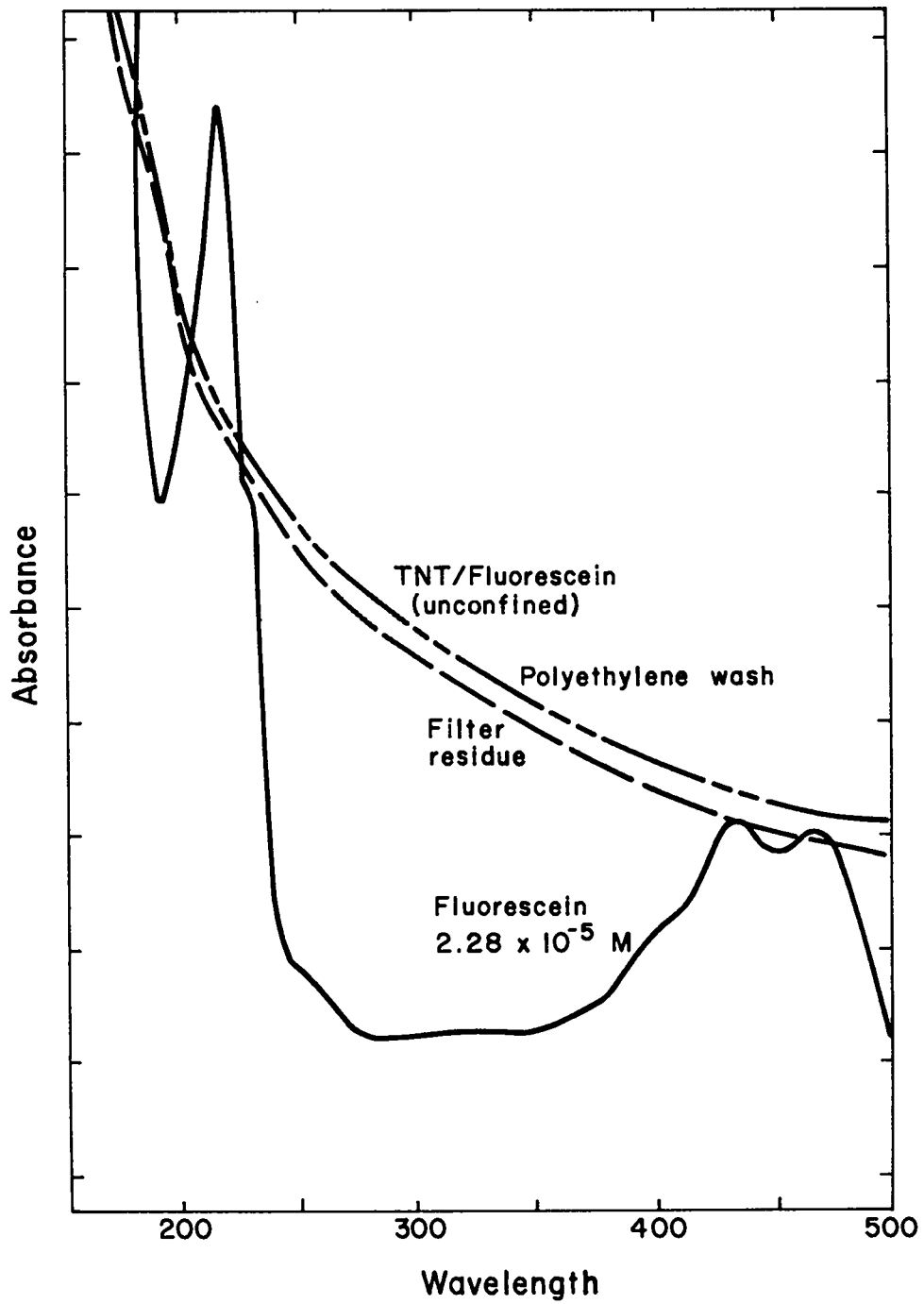


Fig. 4. Spectrophotometric scans of HE residues.

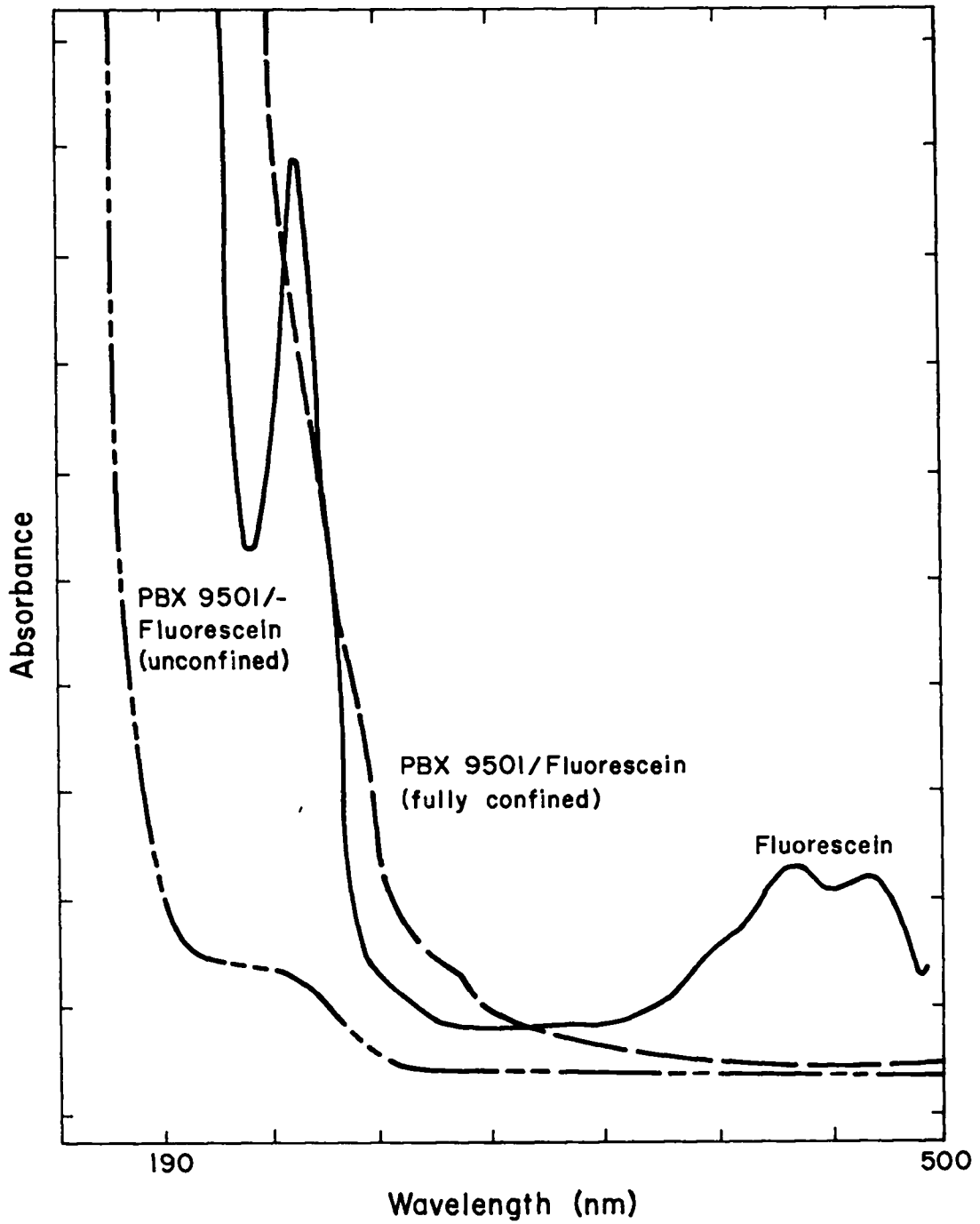


Fig. 5. Spectrophotometric scans of HE residues.

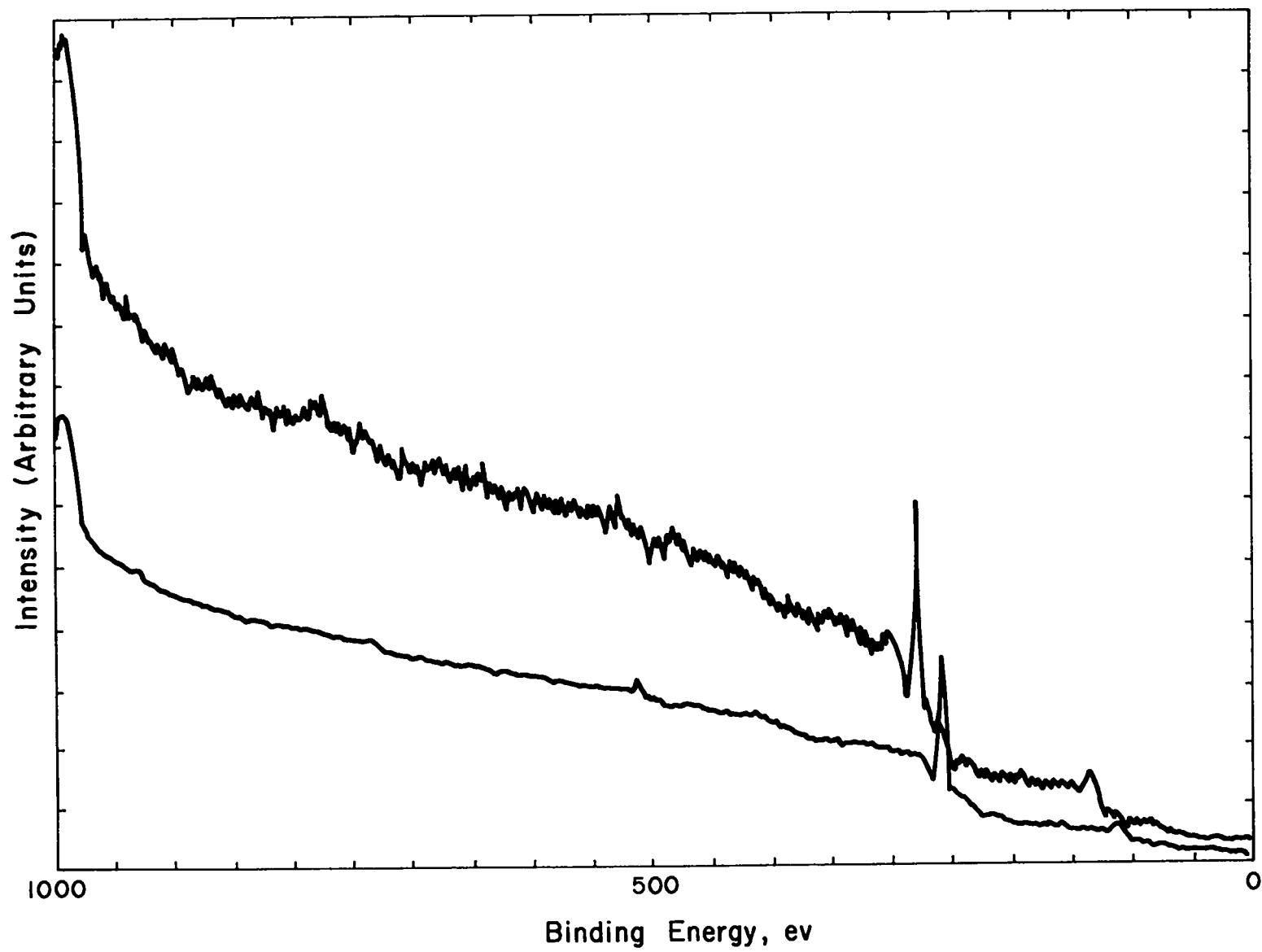


Fig. 6. ESCA scans of HE residues.

evidence of an explosive signature was found for confined detonation in air.

Discussions with several explosives experts indicate that explosive residues would not survive the detonation of an efficiently designed implosion system, and that the gaseous detonation products would be the equilibrium products, because of the oxidizing/reducing effects from surrounding materials and air. Any signature from a combination of equilibrium gaseous products would be lost because of mixing. Our experiments tend to support these statements.

Printed in the United States of America
 Available from
 National Technical Information Service
 US Department of Commerce
 5285 Port Royal Road
 Springfield, VA 22161
 Microfiche \$3.50 (A01)

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