

TECHNICAL NOTE

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Improved Method for the Analysis of the Military Explosive Composition C-4

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ABSTRACT: A procedure for the separation and identification of the major components of the military plastic explosive composition C-4 is described. The explosive (RDX), plasticizer, and rubber binder are separated by selective solvent extraction and filtration, and the extracts are analyzed by multiple internal reflection infrared spectroscopy.

KEYWORDS: forensic science, explosives, spectroscopic analysis, plastic, C-4, multiple internal reflectance, Fourier transform, infrared spectroscopy

The military explosive composition C-4 is a white putty-like deformable material containing the high explosive hexahydro-1,3,5-trinitro-*S*-triazine (RDX). In the examination of explosives evidence, a white deformable material that has been found to contain RDX could be presumed to be C-4. However, other explosives (military and civilian) also contain RDX, and these sources cannot be ruled out until a more definitive analysis is performed. A listing of the ingredients in C-4 is shown in Table 1.

Previous work described the identification of two components in C-4 by transmission infrared spectrophotometry [2,3]. In these studies, extracts containing RDX and plasticizer were analyzed as solutions in liquid cells. The lack of interference from other ingredients was mentioned [2], but their characterizations were not carried out.

A more sensitive and definitive method for the examination of C-4 has been developed. By this method, three of the constituents are separated by a simple solvent extraction and filtration procedure. The separated components are subsequently identified by internal reflection Fourier transform infrared spectroscopy (wavelength dispersive instruments may be used as well).

Background

Multiple internal reflectance (MIR) infrared spectroscopy is a method of choice for the characterization of nonvolatile soluble materials because of its ease of application, high sensitivity, and relative freedom from sample thickness constraints [4]. An extract is applied to

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TABLE 1—*Composition of C-4.*^a

Compound	Weight, %
RDX	91.00
Plasticizer (DOS or DOA)	5.30
Polyisobutylene (PIB)	2.10
Motor oil	1.60

^aSource: Ref 1.

the surface of the MIR crystal and the solvent is allowed to evaporate. A sample thickness of only a few micrometres is enough to produce a usable infrared spectrum. As a result, the amount of sample is considerably less than that required for transmission work.

Chloroform is a good solvent for the separation of plasticizers and binder from C-4 [2,3]. However, the application of a clarified chloroform extract to an MIR crystal can lead to the appearance of interfering RDX absorption bands in the infrared spectrum (see Fig. 1). The RDX bands are not reduced by prolonged centrifugation or microfiltration, and are presumably caused by low levels of dissolved RDX that precipitate directly onto the MIR crystal surface during solvent evaporation. This dissolved RDX is not seen in transmission spectroscopy.

To remove the residual RDX from the plasticizer, as well as separate out the binder, polyisobutylene (PIB), the following procedure was developed.

Equipment, Materials, and Reagents

All infrared spectroscopic analyses were performed on an Analect FX-6250 Fourier transform infrared spectrophotometer. The multiple internal reflectance (MIR) technique was applied to samples deposited on a 52.5- by 20- by 2-mm 45° KRS-5 (thallium bromide-iodide) crystal (Wilks Division of Analabs, Inc., North Haven, CT). Reagent grade acetone, carbon tetrachloride, and chloroform (preserved with 0.5% ethanol) were used in the extraction procedures, and filtrations were carried out on compatible 25-mm membrane filter discs (Gelman Alpha-450 or TF-450) in a glass filter holder. Beakers (50 mL, 100 to 250 mL), a magnetic stirrer, and side arm test tubes were also employed.

Procedure

Extractive Separations

1. Place approximately 50 mg of suspected C-4 in a 50-mL beaker, add 10 mL of chloroform and stir on a magnetic stirrer until the sample is completely dispersed. (If the sample does not disperse, it is not C-4.)
2. Filter solution on a membrane filter disc. The undissolved crystals are RDX. Wash with 3 to 5 mL of chloroform.
3. Pour clear filtrate into an equal volume of acetone and mix. The white cloudy precipitate that develops is PIB.
4. Filter mixture on a fresh membrane filter disc. The filtrate clears and the PIB remains on the filter.
5. Pour filtrate from Step 4 into a 100- to 250-mL beaker and evaporate to dryness over low heat. The residue contains plasticizer and residual RDX, motor oil, and possibly some PIB.
6. Add a few millilitres of carbon tetrachloride to the residue and swirl. Filter on a fresh membrane filter disc. The filtrate contains the plasticizer and motor oil.

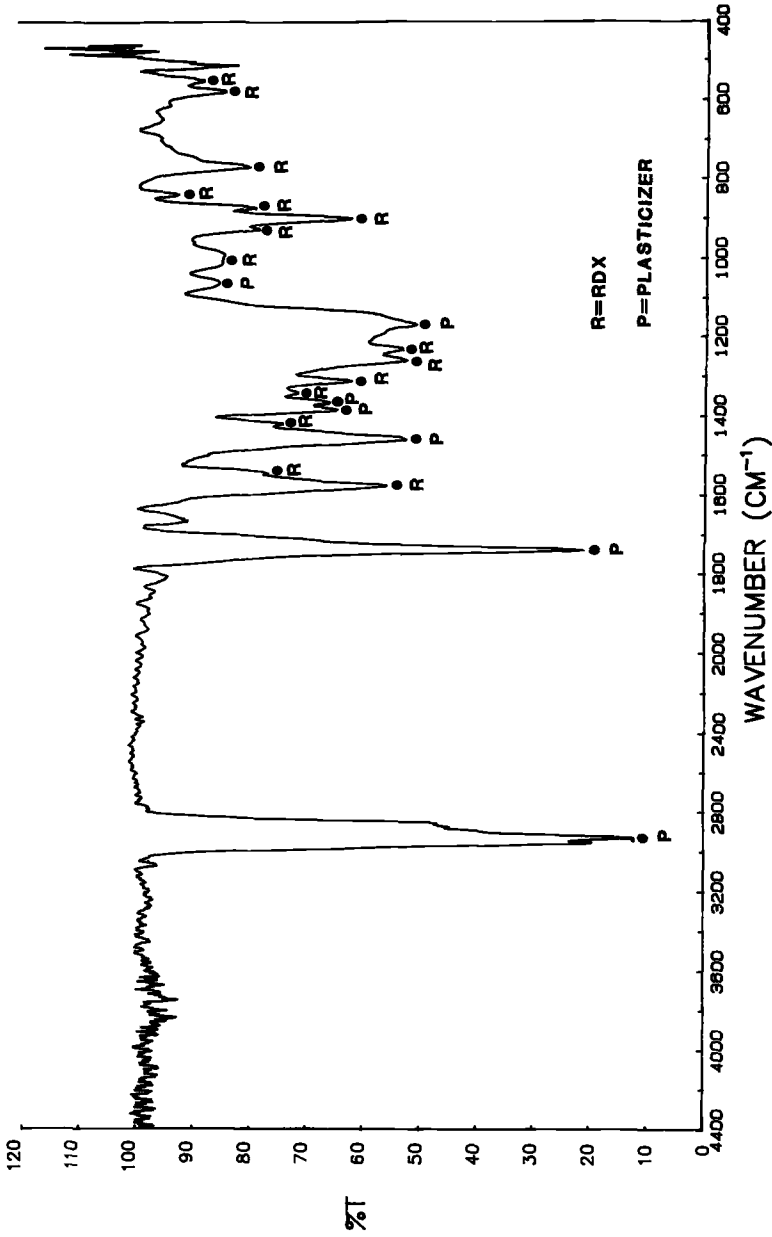


FIG. 1—MIR spectrum of clarified chloroform extract of C-4.

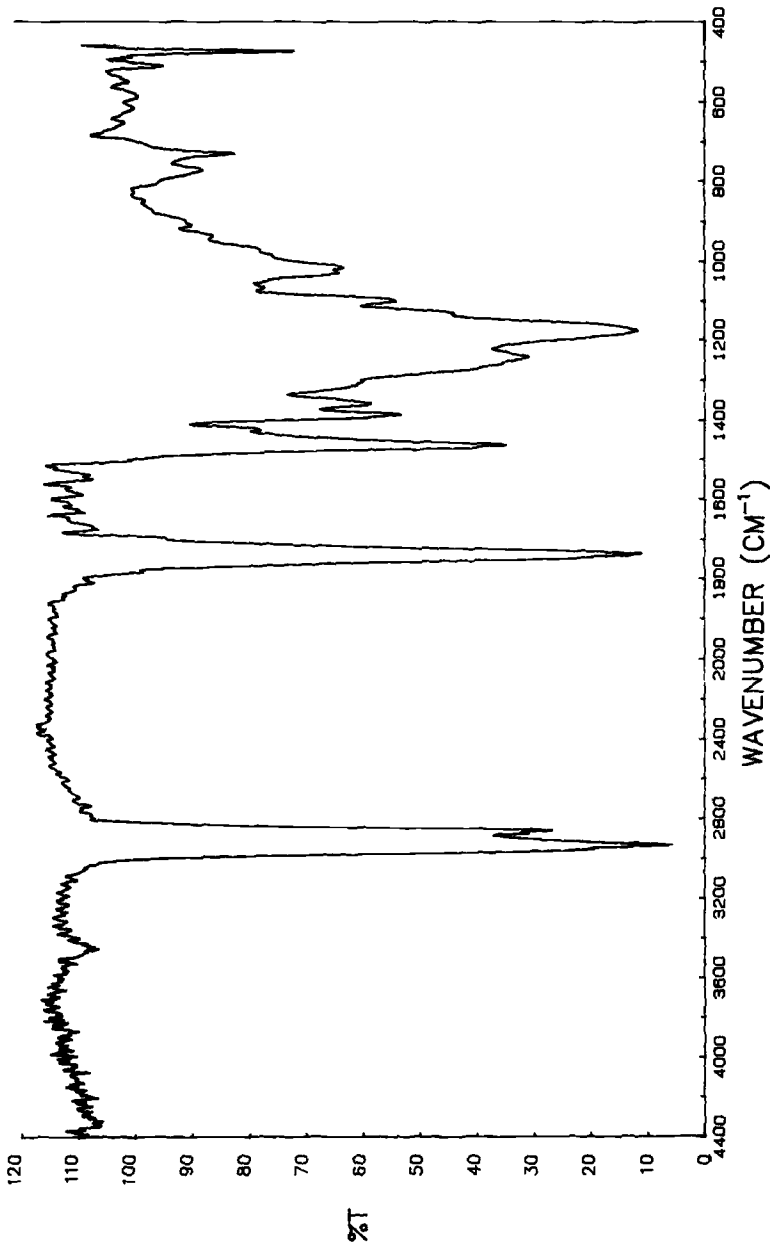


FIG. 2—MIR spectrum of di(2-ethylhexyl) sebacate (DOS).

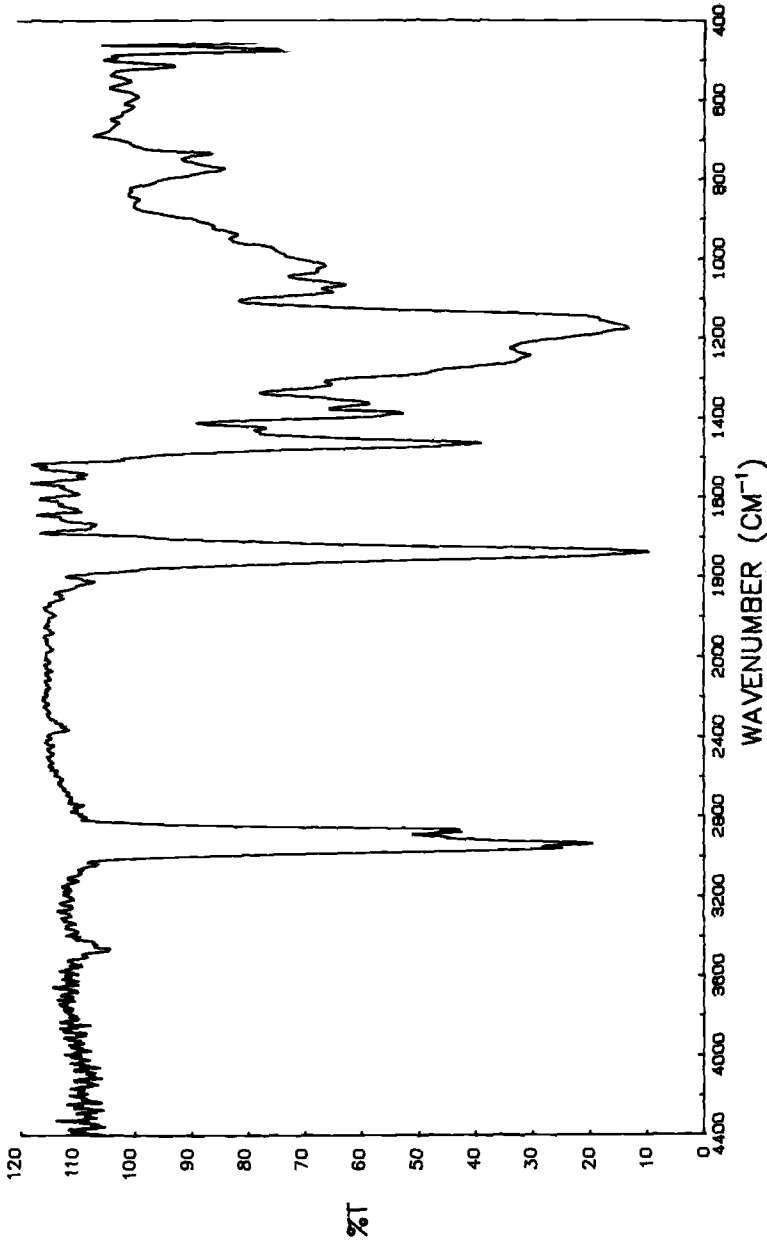


FIG. 3—MIR spectrum of di(2-ethylhexyl) adipate (DOA).

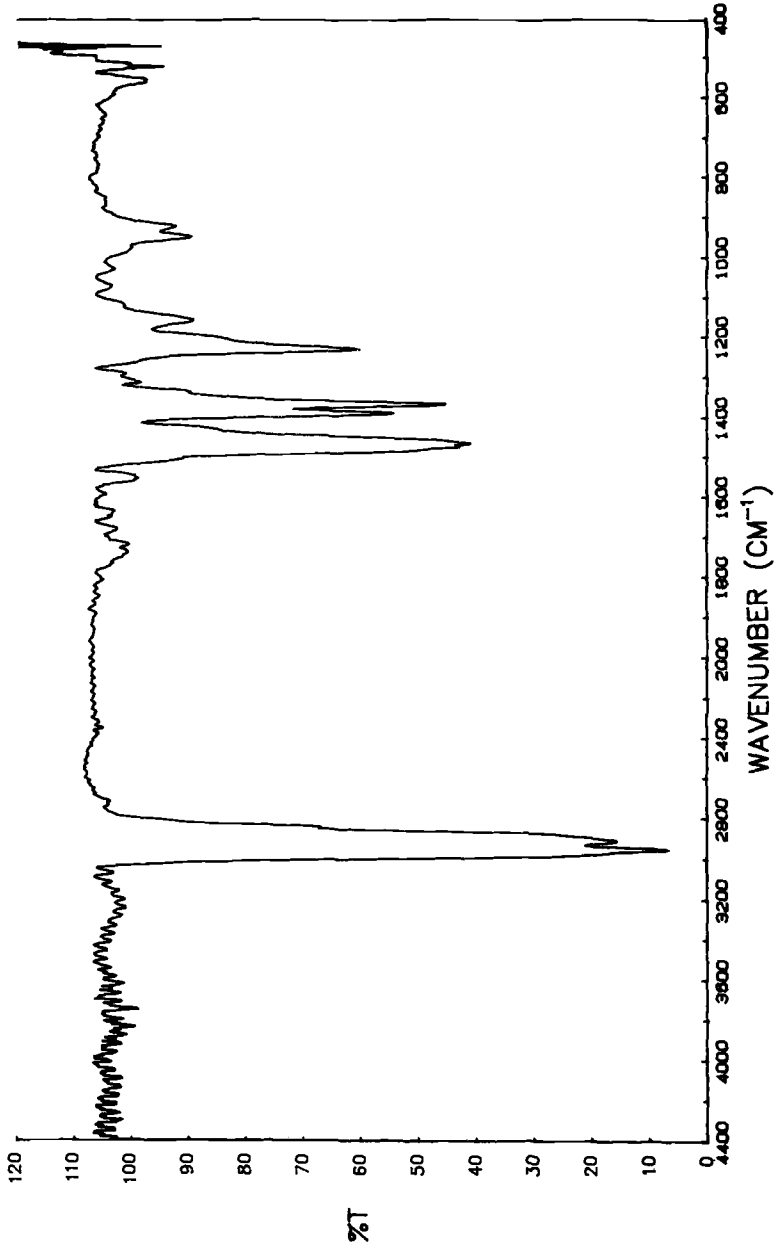


FIG. 4—MIR spectrum of polyisobutylene (PIB).

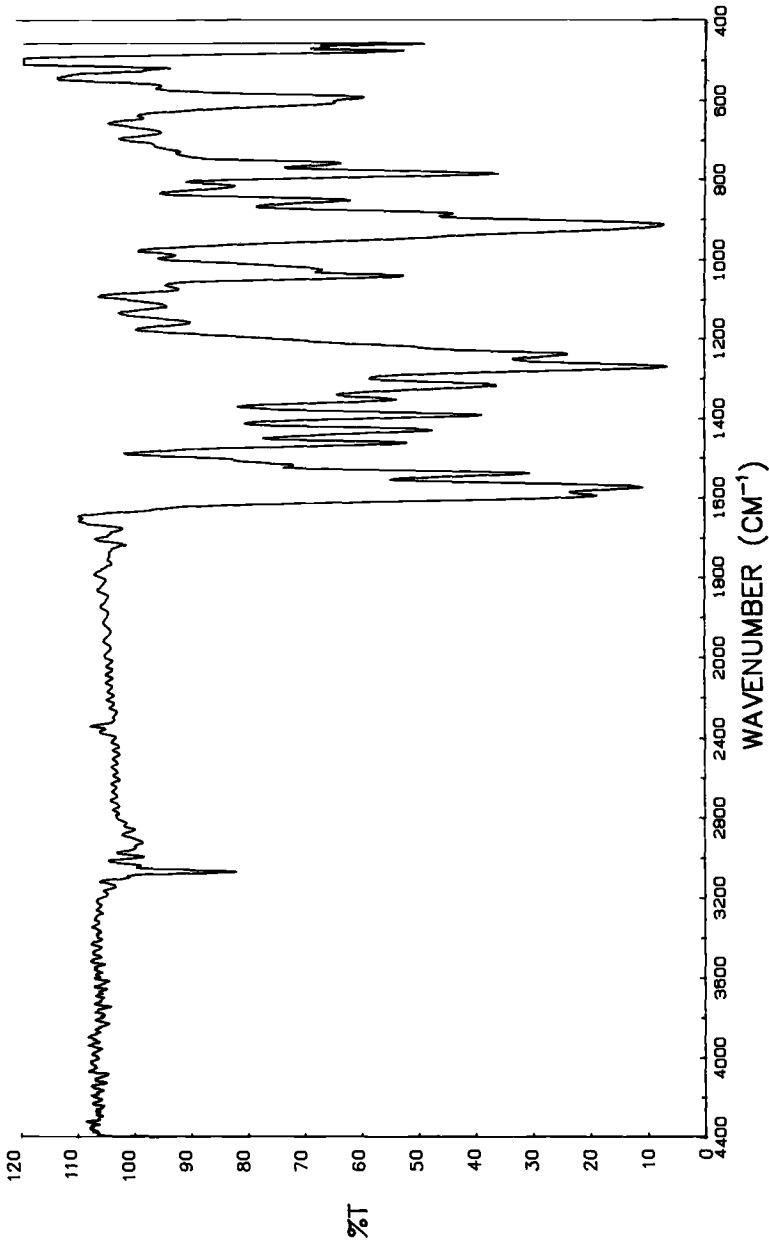


FIG. 5—MIR spectrum of RDX.

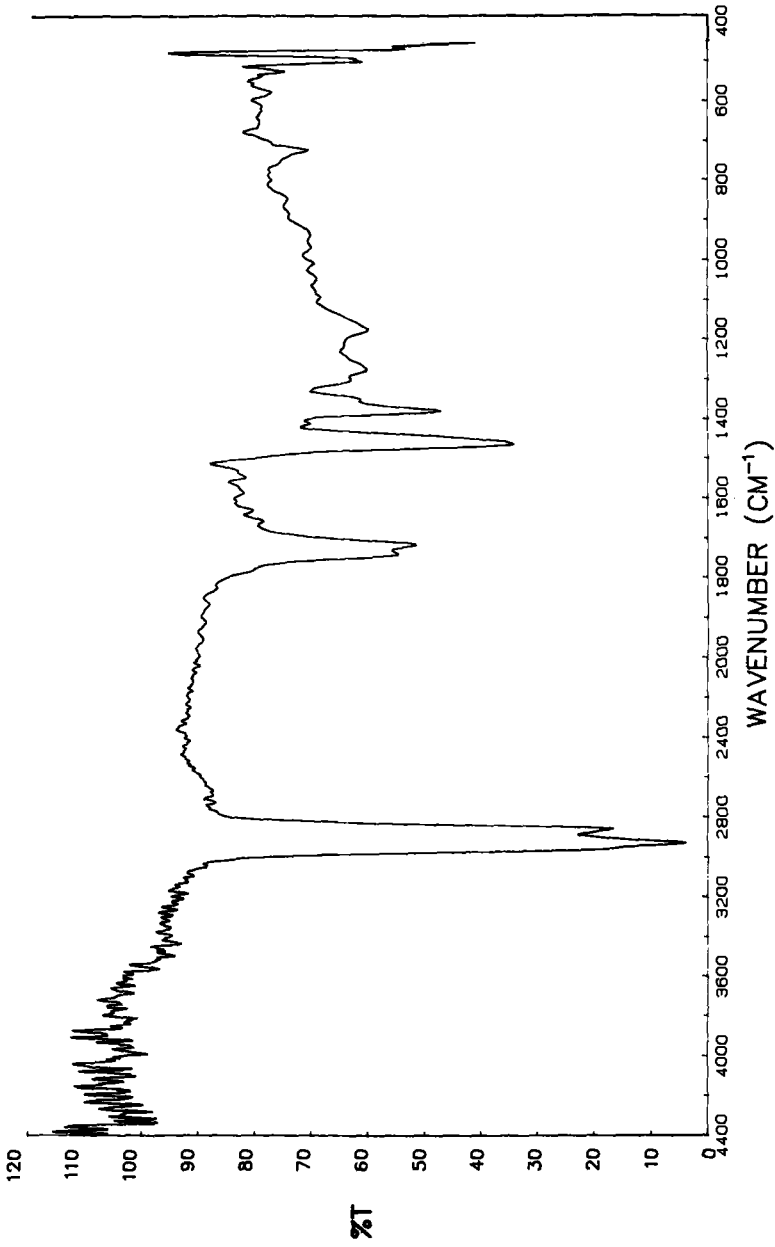


FIG. 6—MIR spectrum of carbon tetrachloride extract of PE-4.

Infrared Spectroscopy

7. Place several drops of the filtrate containing plasticizer (Step 6) on MIR crystal, allow solvent to evaporate, and scan in an infrared spectrophotometer. Representative spectra of di(2-ethylhexyl) sebacate (DOS), and di(2-ethylhexyl) adipate (DOA) are shown in Figs. 2 and 3, respectively. (Motor oil does not interfere substantially.)

8. Place filter disc bearing PIB (Step 4) into a small beaker and add a few millilitres of chloroform and stir to redissolve PIB. Place several drops of this solution onto MIR crystal and allow solvent to evaporate. Rinse surface of crystal with a few millilitres of carbon tetrachloride to remove residual plasticizer that may not have completely separated from PIB in Step 4. Scan MIR crystal in an infrared spectrophotometer. A representative MIR spectrum of PIB is shown in Fig. 4.

9. Dissolve RDX crystals (Step 2) in a few millilitres of acetone, apply to MIR crystal, evaporate solvent, and scan in an infrared spectrophotometer. A representative MIR spectrum of RDX is shown in Fig. 5.

Results

By the use of this procedure, a rapid and indisputable identification of C-4 may be obtained. The lower practicable limit of the sample size needed has not been determined, but is estimated to be in the low milligram range. The use of Fourier transform infrared techniques, specialized MIR attachments and sample application techniques [5], and proportional reductions in the extraction solvent volumes would reduce this limit significantly.

The British plastic explosive PE-4 does not contain PIB and, as a result, will not show a cloudy precipitate in Step 3. Explosive compositions such as this may contain binders that consist of natural fatty acid esters such as bee's wax. If the analysis scheme were carried to completion on PE-4, the infrared spectrum for the carbon tetrachloride extract (Step 6) would look like Fig. 6.

One may go one step further in the analysis of C-4 by separating the motor oil from the plasticizer by gas chromatography. This was not felt to be necessary for the identification of C-4 because of the unique nature of a mixture of the compounds already identified. However, if a comparison of one sample of C-4 to another is desired, gas chromatography may provide a hydrocarbon "signature" for the particular oil used.

References

- [1] Department of the Army Technical Manual TM-9-1300-214/Department of the Air Force Technical Manual TD-11A-1-34 (joint publication), Nov. 1967.
- [2] Midkiff, C. R. and Washington, W. D., "Systematic Approach to the Detection of Explosive Residues. IV. Military Explosives," *Journal of the Association of Official Analytical Chemists*, Vol. 59, No. 6, Nov. 1976, pp. 1357-1374.
- [3] Peimer, R. E., Washington, W. D., and Snow, K. B., "On the Examination of the Military Explosive, C-4," *Journal of Forensic Sciences*, Vol. 25, No. 2, April 1980, pp. 398-400.
- [4] Harrick, N. J., *Internal Reflection Spectroscopy*, John Wiley & Sons, New York, 1967.
- [5] Gilley, A. C., Cassels, J., and Wilks, P. A., Jr., "Internal Reflection Spectroscopy. III. Micro Sampling," *Applied Spectroscopy*, Vol. 24, No. 5, Sept./Oct. 1970, pp. 539-543.

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