

TECHNICAL NOTE

*R. J. Kopec,¹ B.S.; W. D. Washington,¹ B.S.; and
C. R. Midkiff, Jr.,¹ M.S.*

Forensic Applications of Sapphire Cell-Infrared Spectroscopy: Companion to the Diamond Cell in Explosive and Leg Wire Identification

The application of the diamond cell sample holder to the infrared examination of trace evidence has been reported by several investigators. Materials examined by this technique have included paints [1-4], blasting cap leg wire insulation [5], and explosives [6, 7]. With the diamond cell, the usable infrared spectral region is approximately 1800 to 200 wave numbers (cm^{-1}). While this region allows the characterization of many materials, the analyst may desire examination of the range 4000 to 200 cm^{-1} . With a sapphire cell, the region 4000 to 1600 cm^{-1} , which is partially opaque to the diamond, can be used to obtain complete coverage of the desired working range [8]. A sapphire window sample holder compatible with the diamond cell holder mount is commercially available² and is appreciably less expensive than the diamond cell. A blank spectrum obtained with the sapphire cell is shown in Fig. 1. In practice, a sample can be transferred manually from

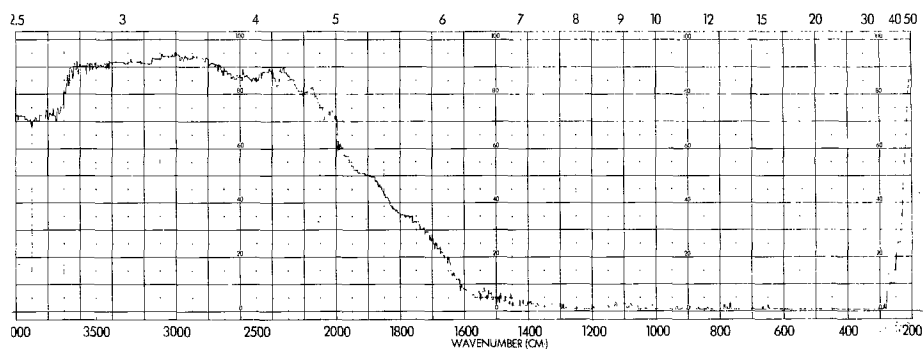


FIG. 1—Infrared spectrum of sapphire cell.

Received for publication 23 March 1977; accepted for publication 3 June 1977.

¹Forensic scientist and forensic chemists, respectively, Bureau of Alcohol, Tobacco and Firearms, U.S. Treasury Department, Washington, D.C. 20226.

²High Pressure Diamond Optics, Inc., McLean, Va.

the diamond cell to the sapphire cell to obtain the total infrared spectrum. All spectra were obtained on a Perkin-Elmer Model 621 Grating Infrared Spectrophotometer equipped with a $4\times$ all-reflecting beam condenser and sapphire cell sample holder. An unattenuated signal of 4 to 6% was attenuated in the reference beam to 75 to 80% transmittance. Instrument operating settings and parameters were slits, 1100; gain, 8; attenuator speed, 1000; scan time, 10 min; suppression, 0; source current, 0.8A; and slits at $2\times$ programmed. When the sapphire cell is used excessive pressure should be avoided since the softer window material is more easily fractured than the diamond.

To evaluate the forensic applications of the sapphire cell, spectra of blasting cap leg wire insulation materials and explosives were obtained and compared with film, solution, and KBr pellet spectra of the same materials. In addition to the expanded spectral range, the discriminating value of the sapphire cell spectra alone was examined. Spectra of the major types of electric blasting cap leg wire insulation materials from 4000 to 1600 cm^{-1} are shown in Figs. 2 to 7. The inner curves in these spectra represent attenuation adjust-

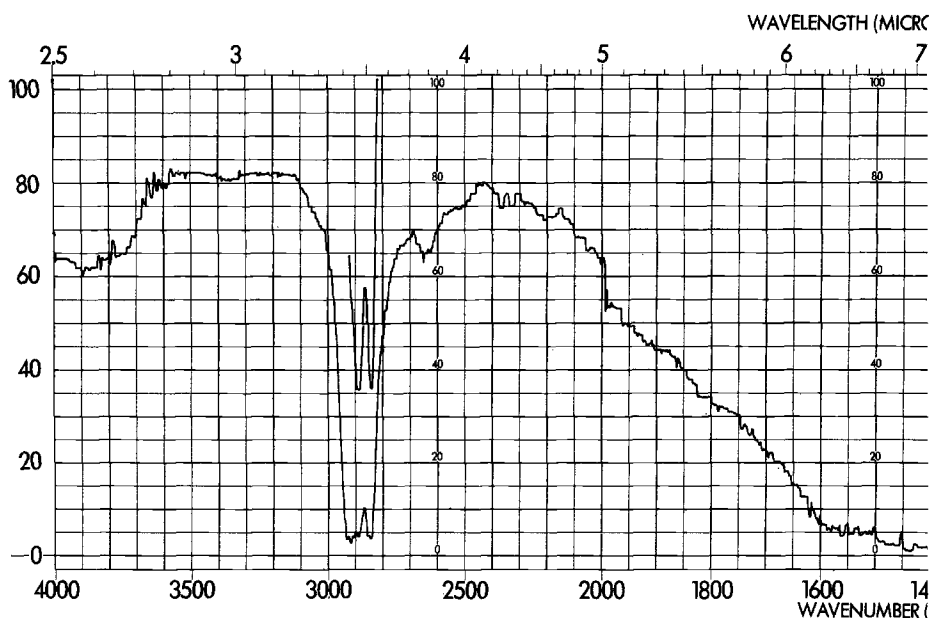


FIG. 2—High density polyethylene Du Pont insulation.

ments to improve resolution. A spectrum of polystyrene is shown in Fig. 8. Similar spectra of four representative explosives are shown in Figs. 9 to 12. Since domestic cyclotrimethylenetrinitramine (RDX) can contain up to 10% cyclotetramethylenetetranitramine (HMX), a spectrum of 5% HMX in RDX is shown in Fig. 13.

Results and Discussion

Inspection of Figs. 2 to 5 shows that, from the sapphire cell spectra alone, the major types of leg wire insulation can be readily identified as to material type and distinguished from each other. With nylon (Fig. 6) and Teflon® (Fig. 7) discrimination is difficult; however, both may be distinguished from the other types. For differentiation of nylon and Teflon, the sample may be examined in the diamond cell. Teflon has two strong bands at 1155 and 1210 cm^{-1} that are absent in nylon [5].

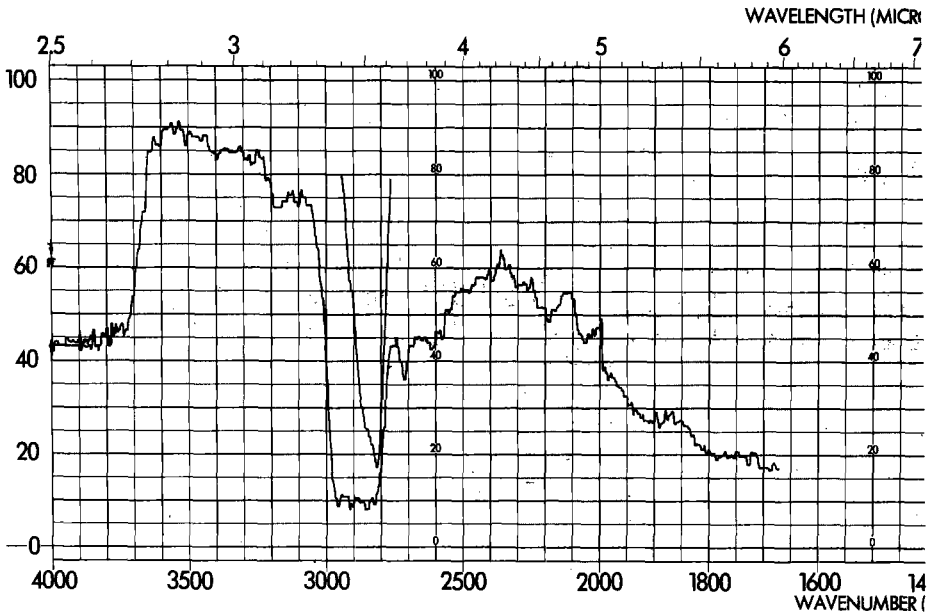


FIG. 3—Polyethylene/polypropylene Hercules insulation.

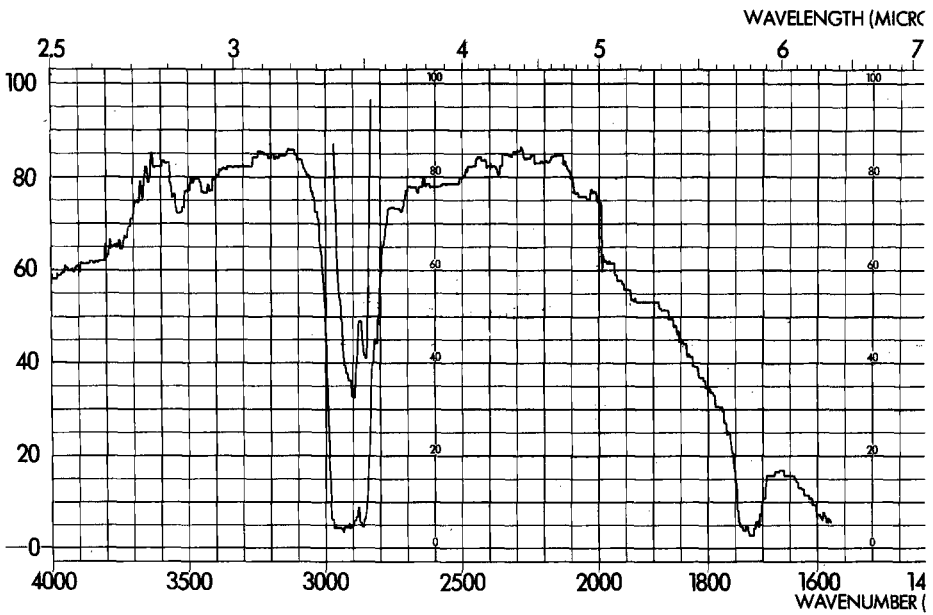


FIG. 4—Polyvinylchloride Atlas insulation (also ICI imported by Austin Powder Co.).

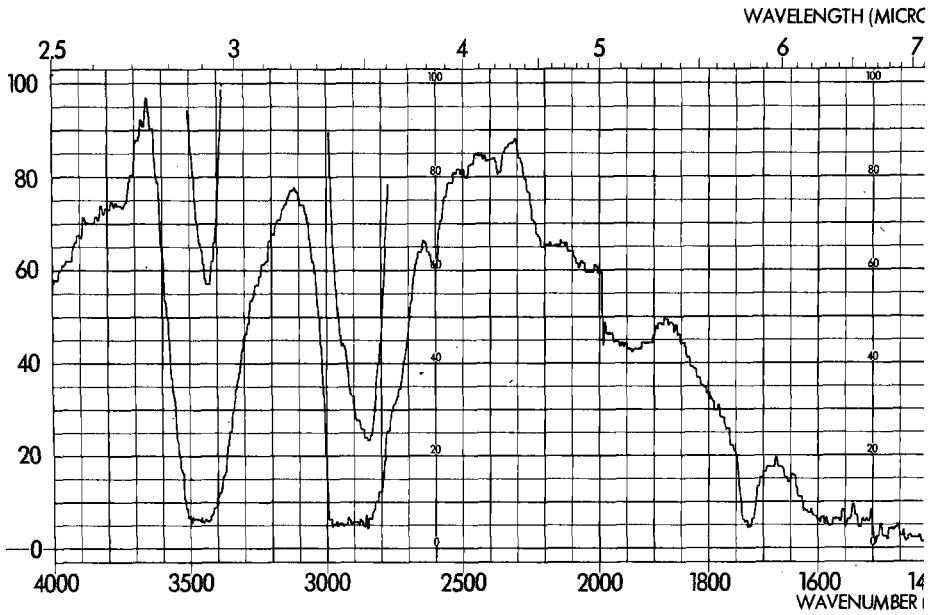


FIG. 5—Ethyl cellulose Hercules (military) insulation.

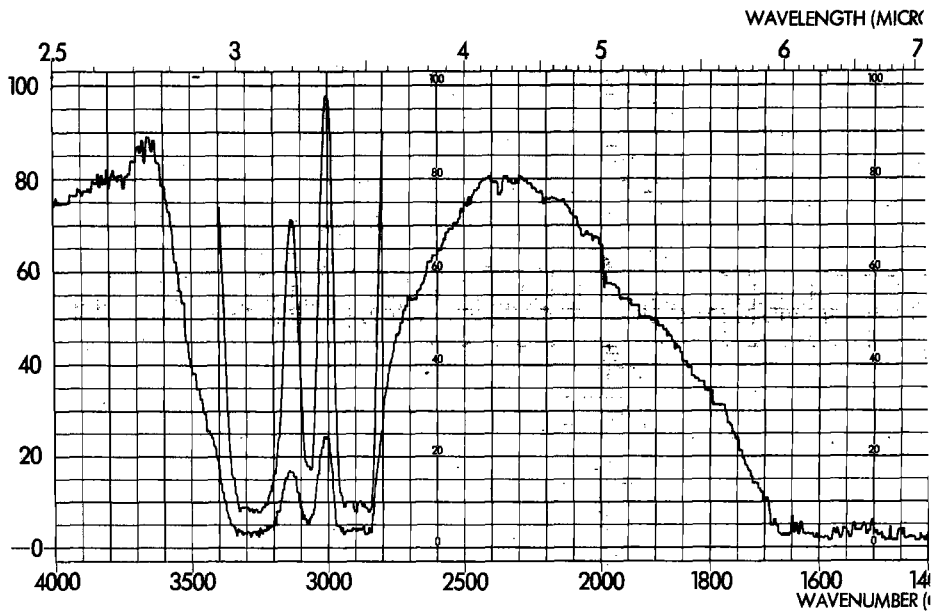


FIG. 6—Nylon Du Pont insulation.

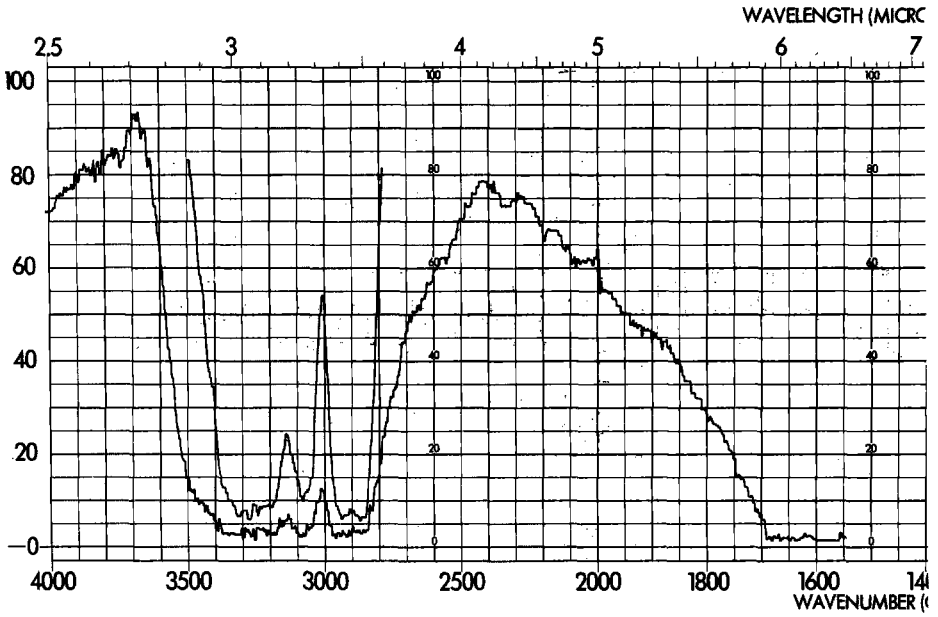


FIG. 7—Teflon Du Pont insulation.

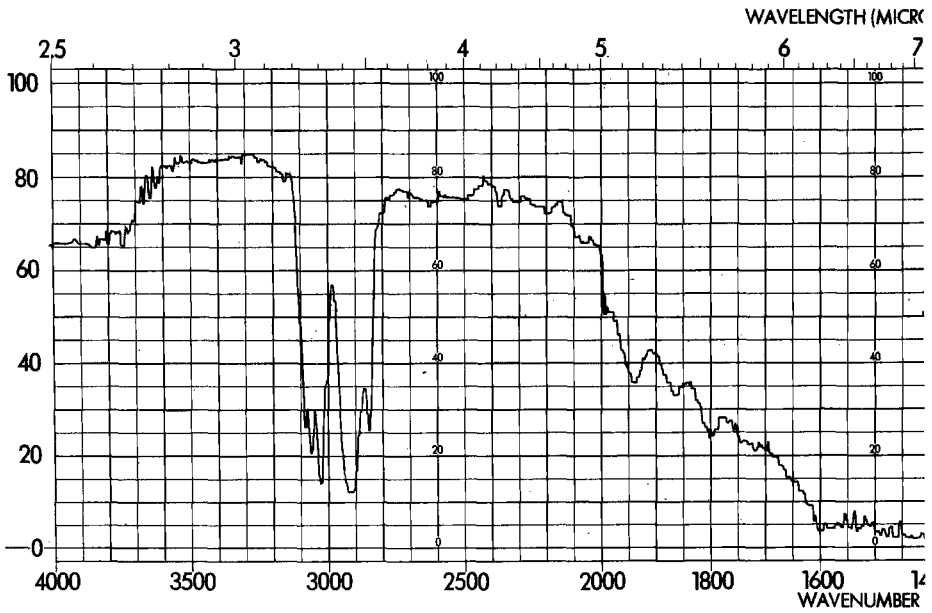


FIG. 8—Polystyrene calibration.

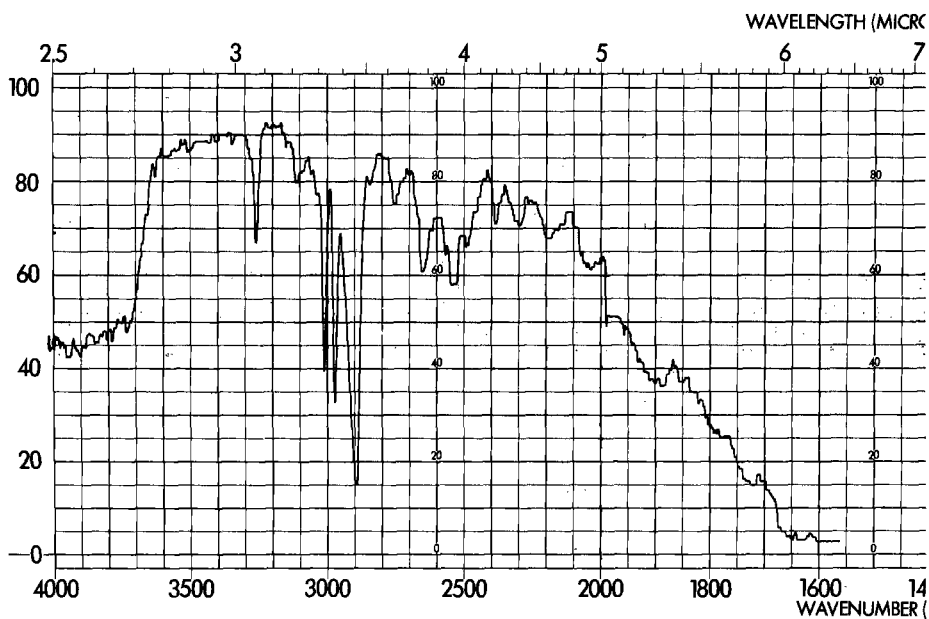


FIG. 9—Pentaerythritol tetranitrate (PETN).

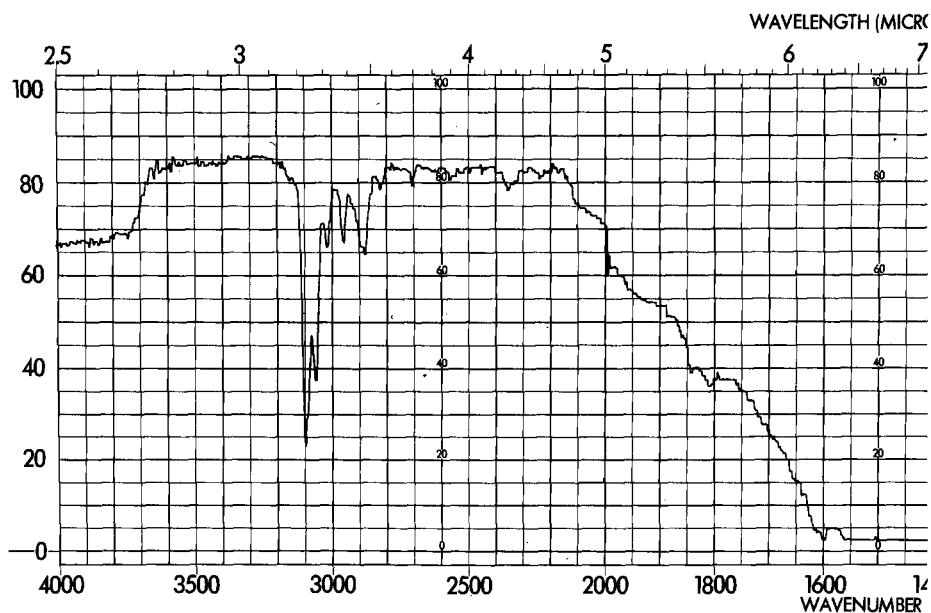


FIG. 10—2,4,6-Trinitrotoluene (TNT).

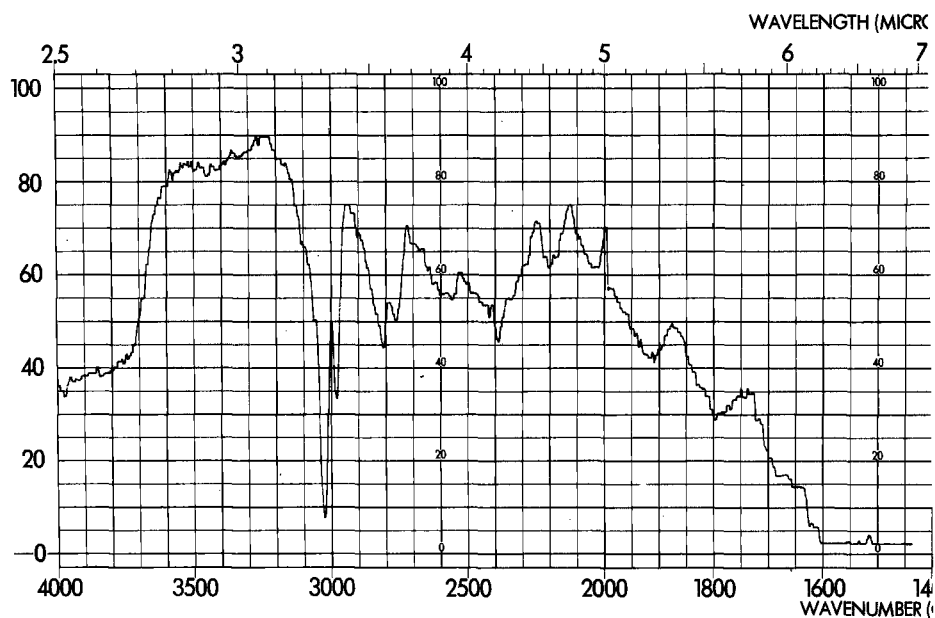


FIG. 11—Cyclotetramethylenetetranitramine (HMX).

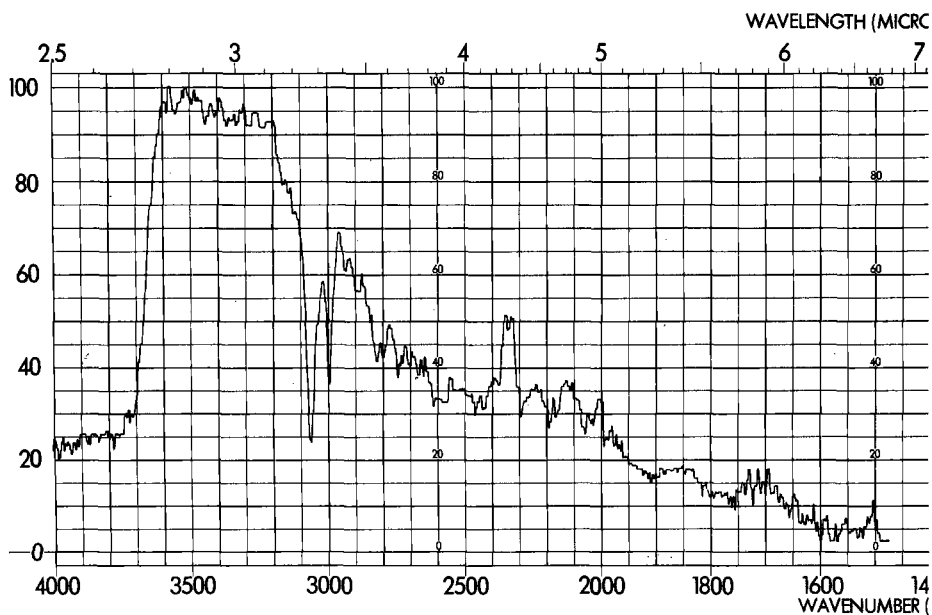


FIG. 12—Cyclotrimethylenetrinitramine (RDX).

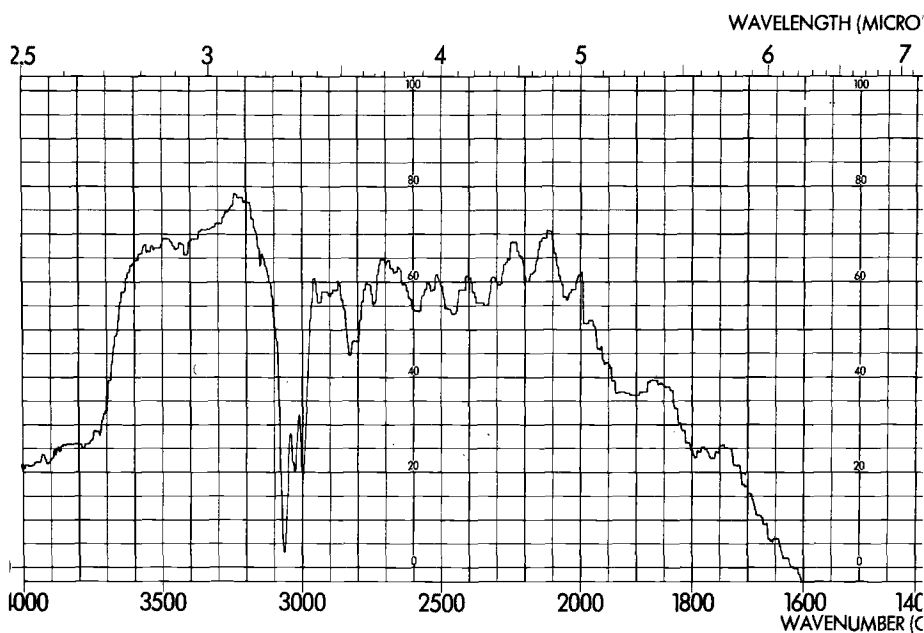


FIG. 13—Five percent mixture of HMX in RDX.

With the four explosives shown in Figs. 9 to 12 little difficulty is encountered in discrimination based on the sapphire cell spectra alone. Further work is required to determine if these spectra alone are sufficient for compound identification because, over the range examined, other explosives or materials may have spectra sufficiently similar to preclude unique identification. The use of the sapphire cell in conjunction with the diamond cell provides spectral coverage adequate for explosive identification. In Fig. 13, the band at 3020 cm^{-1} attributable to HMX is readily seen. This amount of HMX can also be detected in RDX with the diamond cell; however, with either solution or KBr pellet spectra, its detection is quite difficult. The ability to detect a small quantity of an additional component(s) in RDX can be of value in the comparison of known and questioned samples in the investigation of bombings or seizures of military explosives [6].

Conclusion

While it may have somewhat limited application in the forensic laboratory, the sapphire cell has considerable potential. For the laboratory with limited funds, the sample holder with sapphire cells could be purchased initially and diamond cells added later. This would provide a significant enhancement of infrared analysis capability, particularly for the examination of the small samples typical of trace evidence.

References

- [1] Tweed, F. T., Cameron, R., Deak, J. S., and Rodgers, P. G., "The Forensic Microanalysis of Paints, Plastics and Other Materials by an Infrared Diamond Cell Technique," *Forensic Sciences*, Vol. 4, No. 3, Nov. 1974, pp. 211-218.
- [2] Rodgers, P. G., Cameron, R., Cartwright, N. S., Clark, W. H., Deak, J. S., and Norman, E. W. W., "The Classification of Automobile Paint by Diamond Window Infrared Spectrophotometry. Part I: Binders and Pigments," *Canadian Society of Forensic Science Journal*, Vol. 9, No. 1, March 1976, pp. 1-14.

- [3] Rodgers, P. G., Cameron, R., Cartwright, N. S., Clark, W. H., Deak, J. S., and Norman, E. W. W., "The Classification of Automobile Paint by Diamond Window Infrared Spectrophotometry. Part II: Automotive Topcoats and Undercoats," *Canadian Society of Forensic Science Journal*, Vol. 9, No. 2, June 1976, pp. 49-68.
- [4] Rodgers, P. G., Cameron, R., Cartwright, N. S., Clark, W. H., Deak, J. S., and Norman, E. W. W., "The Classification of Automobile Paint by Diamond Window Infrared Spectrophotometry. Part III: Case Histories," *Canadian Society of Forensic Science Journal*, Vol. 9, No. 3, Sept. 1976, pp. 103-111.
- [5] Washington, W. D. and Midkiff, C. R., "Forensic Applications of Diamond Cell-Infrared Spectroscopy. I: Identification of Blasting Cap Leg Wire Manufacturers," *Journal of Forensic Sciences*, Vol. 21, No. 4, Oct. 1976, pp. 862-867.
- [6] Midkiff, C. R., Jr. 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.
- [7] Washington, W. D., Kopec, R. J., and Midkiff, C. R., Jr., "Systematic Approach to the Detection of Explosive Residues. V. Black Powders," *Journal of the Association of Official Analytical Chemists*, Vol. 60, No. 6, Nov. 1977, pp. 1331-1340.
- [8] Lippincott, E. R., Welsh, F. F., and Weir, C. E., "Microtechnique for the Infrared Study of Solids: Diamonds and Sapphires as Cell Materials," *Analytical Chemistry*, Vol. 33, No. 1, Jan. 1961, pp. 137-143.

Address requests for reprints or additional information to
Robert J. Kopec
Forensic Branch
Bureau of Alcohol, Tobacco and Firearms
U.S. Treasury Department
Washington, D.C. 20226