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Application of Fourier Transform- Infrared Spectroscopy (FT-IR) for Materials Analysis

by
Philip Patterson and William Lum

Report Date
August 1992



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13. ABSTRACT (Maximum 200 words) In the past, a number of Mission Related Requests have been made to the Coatings, Corrosion, and Packaging Team, Advanced Technology Division at BRDEC to analyze a variety of materials for chemical composition. This technical report presents an overview of one approach, Fourier Transform-Infrared Spectroscopy (FT-IR), the Laboratory Analyst utilizes to support such requests.				
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Preface

For several years, the Coatings and Corrosion Research Team, Advanced Technology Division of the Product Assurance and Engineering Directorate, Belvoir Research, Development and Engineering Center (BRDEC), has utilized infrared spectroscopy for studying polymeric materials used in various Army field support applications. This report presents an overview of this instrumental technique, as well as cites specific application cases. The infrared analyses were quick, non-destructive, and elucidated valuable information regarding the characteristics of the material.

Section I

Background

Infrared spectroscopy has traditionally been recognized as a valuable analytical technique for the identification and characterization of organic compounds. The resulting infrared spectrum is generated through the absorption of infrared radiation by the sample within a given frequency range. Because infrared spectroscopy involves energy transitions at their molecular level, a characteristic spectrum, unique to the material analyzed, is obtained. Thus, the technique is well suited for the qualitative analysis of polymers utilized in coatings, plastics, adhesives, elastomers, and composites.

Section II

Instrumentation

The infrared analyses performed by the Coatings, Corrosion, and Packaging Team are conducted on a Nicolet 5DXB Fourier Transform-Infrared Spectrophotometer (FT-IR). The instrument is equipped with a high intensity, stabilized light source (spectral wavelength range: 4800-400 cm^{-1}), a multi-layered germanium coated KBR beamsplitter, and a deuterated triglycerine sulfate (DTGS) detector. The data system is based on Nicolet's 1280 microprocessor optimized for real-time digitalized data acquisition, spectrophotometer system control, and fast Fourier-Transform processing. This arrangement allows for the use of spectral enhancement routines when a sample is analyzed such as baseline correction, spectral subtraction, reference library searches, and quantitative analysis. Spectral data can be stored using a double-density, dual-sided, 1-megabyte floppy disk.

Section III

Applications

One example of the successful application of FT-IR is the characterization of an unknown polymeric material used as an absorbent in stabilizing environmentally hazardous hydrocarbon spills. The sample was a white, free-flowing particulate solid, which could float on top of water without dissolving. To facilitate the infrared analysis, the material had to be transformed into a cast film. The technique of casting a film from a volatile organic solvent compatible with the sample was used. The material readily dissolved in toluene, so a dilute solution, approximately 10 to 20 percent by weight, was prepared. A few drops of the solution were spread evenly across a potassium bromide crystal (infrared transparent) and oven dried to remove the solvent. The thin polymer film was then analyzed utilizing the routine absorbance/percent transmittance operating mode. The resulting spectrum (Figure 1) indicated that the material was not compositionally similar to the common polymers used in Army coatings (i.e., alkyds, epoxies, cellulose, polyurethanes). By performing the Aldrich-Nicolet FT-IR library search function, a very good matching spectrum of poly (styrene/butadiene) copolymer (Figure 2) was quickly retrieved. A tedious, time consuming review of spectral reference literature was avoided by the successful application of this FT-IR data station software routine. Additionally, the spectrum was stored on a floppy disk as a reference if similar materials are encountered in the future.


Another application of the FT-IR spectrometer is the capability of the instrumentation to perform "non-chemical" separations of components in mixtures. This subtractive technique is often called *spectral stripping* or *difference spectroscopy* and is possible because of the FT-IR's photometric scale accuracy as well as its wave number scale reproducibility when running multiple samples. This technique can be used, for example, in the separation and identification of diluents in epoxy resins. These epoxy resin diluents are added to achieve a certain property and are often classified as either *reactive* or *non-reactive*. Reactive diluents contain the functionally active epoxy

group which becomes an integral part of the crosslinked polymer when cured. Non-reactive diluents, often added as plasticizers to increase film flexibility, contain no such functionally active group and therefore do not add to the crosslinking density. The property of crosslinking density is important because it relates to the film's ability to resist chemical and thermal attack.

The infrared spectrum of an undiluted bisphenol-A epoxy resin, typical of the type required in military primer coating specifications, is shown in Figure 3. The resin was prepared for infrared analysis by casting a thin, uniform film onto a potassium bromide crystal. The spectra of two adulterated epoxy resins prepared in the same manner are shown in Figures 4 and 5.

Utilizing the FT-IR's digital subtraction subroutine, the difference spectra (adulterated resin - "pure" resin * scaling factor) are shown in Figures 6 and 7. Clearly, these spectra represent two different types of compounds.

Figure 6 shows absorbance bands at 1255 cm⁻¹, 915 cm⁻¹, and 850 cm⁻¹ which are characteristic of an epoxy (C⁰-C) ether group. The intense band at 1100 cm⁻¹ is due to the asymmetrical stretch of an alkyl ether group (C-O-C). Also, the strong absorbance bands due to C-H stretching (2975-2850 cm⁻¹) and CH₂ bending (1465 cm⁻¹) indicate the presence of a long chain aliphatic hydrocarbon group. This spectrum matched well to a reference spectra of C12-C14 aliphatic glycidyl ether, a commonly used epoxy reactive diluent.

Figure 7 shows an intense carbonyl (-C=O) band at 1725 cm⁻¹. Also, the bands at 1255 cm⁻¹ and 1125 cm⁻¹ are stretching vibrations due to the presence of an aromatic ester group (O-C=O). The weak absorption bands at 1600 cm⁻¹ and 1510 cm⁻¹ are due to the ^{OR}unsaturated C=C stretching of a ringed nucleus (), confirming the aromaticity of the molecule. The

strong absorbance band at 745 cm^{-1} is due to the out of plane bending of four adjacent hydrogens on an aromatic ring, thereby indicating an ortho-disubstituted compound. Additionally, the bands located from 2960-2870 cm^{-1} are indicative of C-H stretching due to various aliphatic hydrocarbon groupings. This spectrum compared favorably to a reference spectra of dibutylphthalate, a frequently encountered non-reactive plasticizer.

The FT-IR's ability to perform "non-chemical" separations, as in this case of diluents in epoxy resins, significantly assists the analyst in the rapid identification of matrix components.

Attenuated total reflectance (ATR) or multiple internal reflectance (MIR) is another technique frequently used by the infrared spectroscopist. In this technique, the sample is placed in intimate contact with a highly refractive, I. R. transparent crystal (zinc selenide) with a 45 degree interface angle. This positioning allows the incoming infrared radiation to be internally reflected throughout the length of the crystal. These reflections create an evanescent wave which penetrates into the sample/crystal interface (Figure 8). When the sample absorbs this incident radiation, an infrared spectrum is generated. Because ATR allows for sampling depths of only a few microns per reflection, the technique is well suited for studying strongly absorbing materials such as plastics, adhesives, oils, greases, and coating films.

For example, a waste sample of a highly viscous lubricant was analyzed utilizing this technique. The resulting ATR spectrum, shown in Figure 9, was easily interpreted and identified as an aliphatic hydrocarbon based oil.

Section IV

Conclusion

In summary, Fourier Transform-Infrared Spectroscopy has heightened the basic analytical capability of the materials testing and research laboratory. FT-IR has proven to be a potent and cost effective method for the characterization of polymeric compounds found in various military applications. Knowing the nature and composition of these materials advances the tech-base for the formulation and development of higher quality, better performing end use items.

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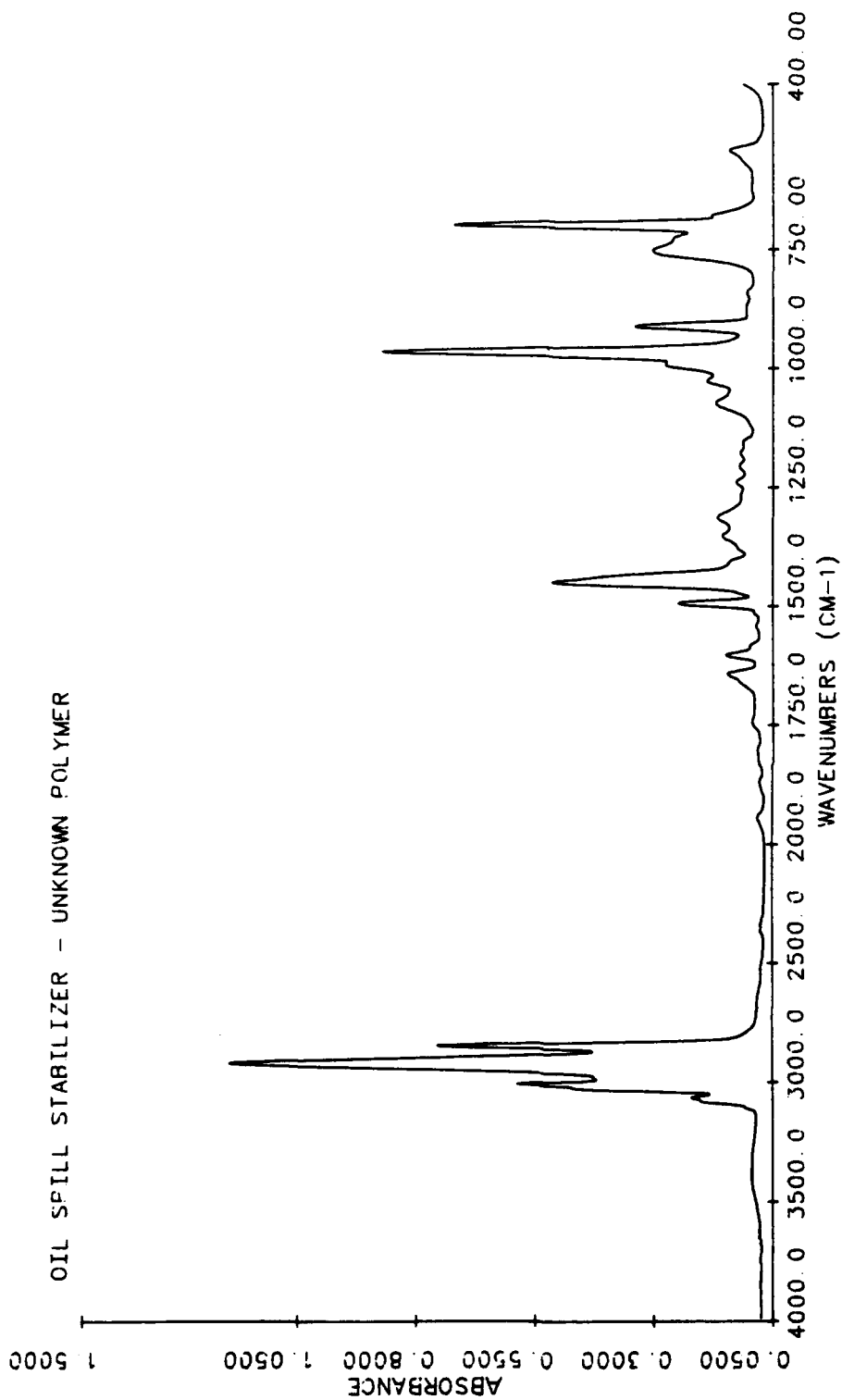


Figure 1. Spectrum of Unknown Polymer

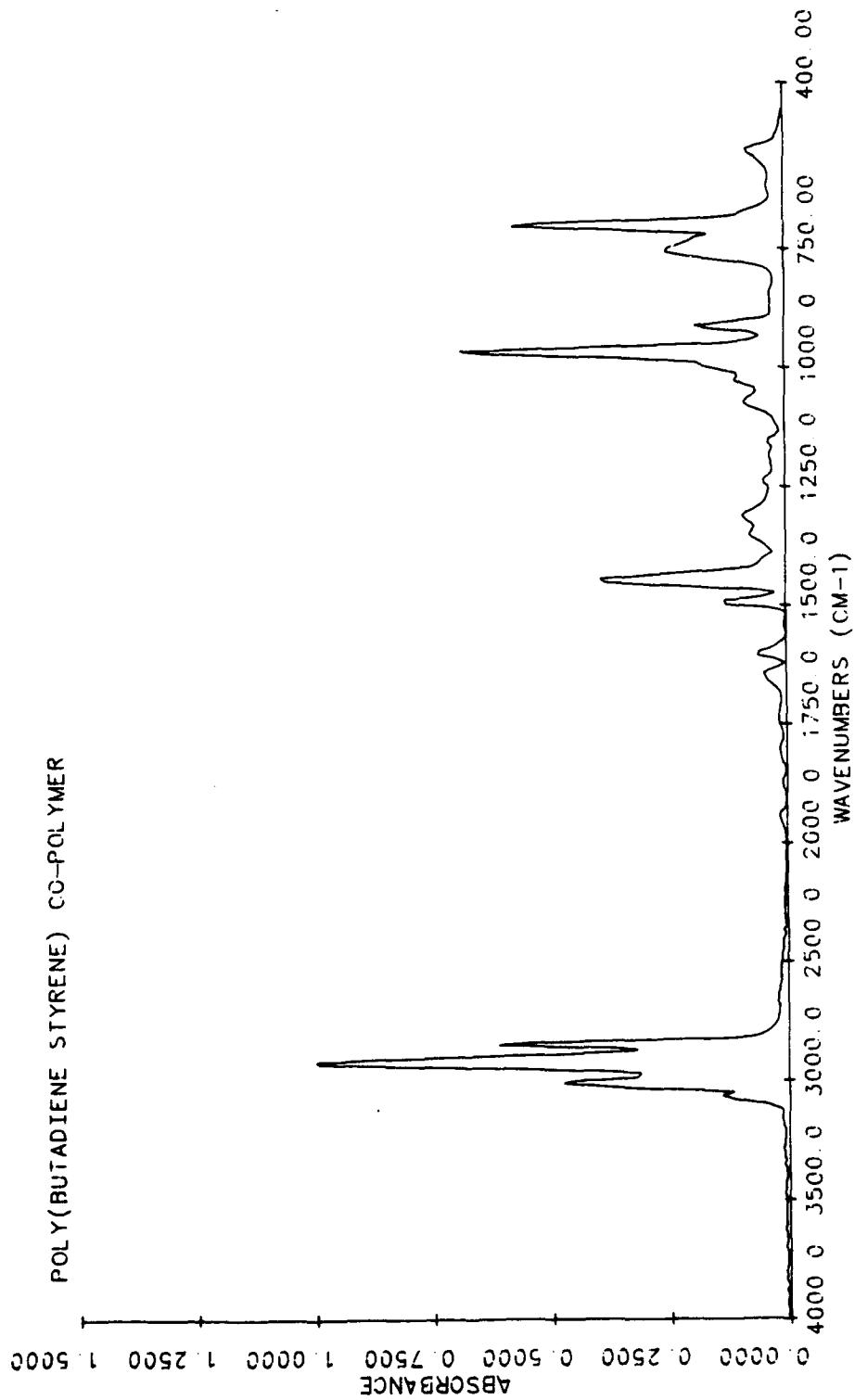


Figure 2. Library Search Spectrum

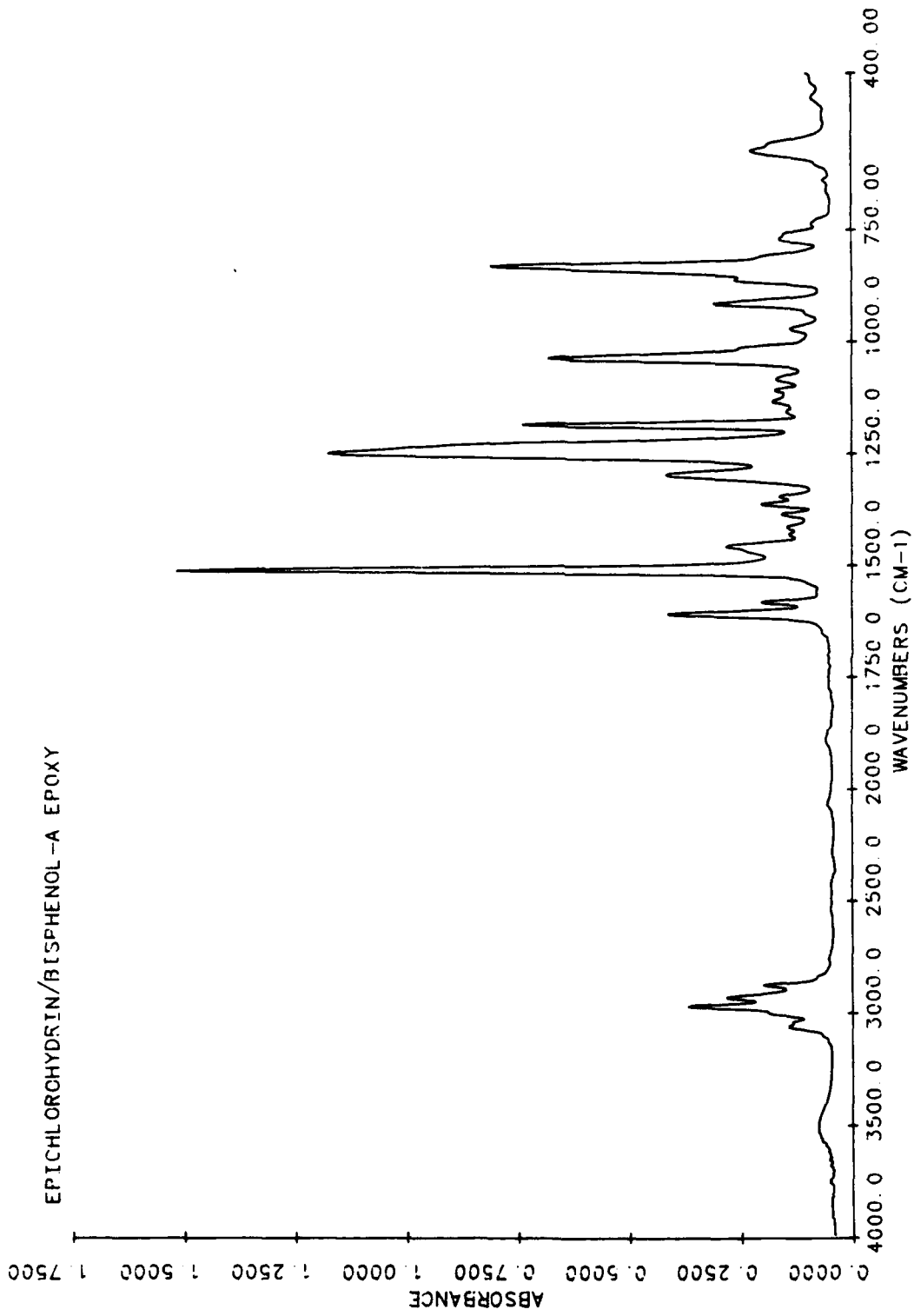


Figure 3. Spectrum of Unmodified Epoxy

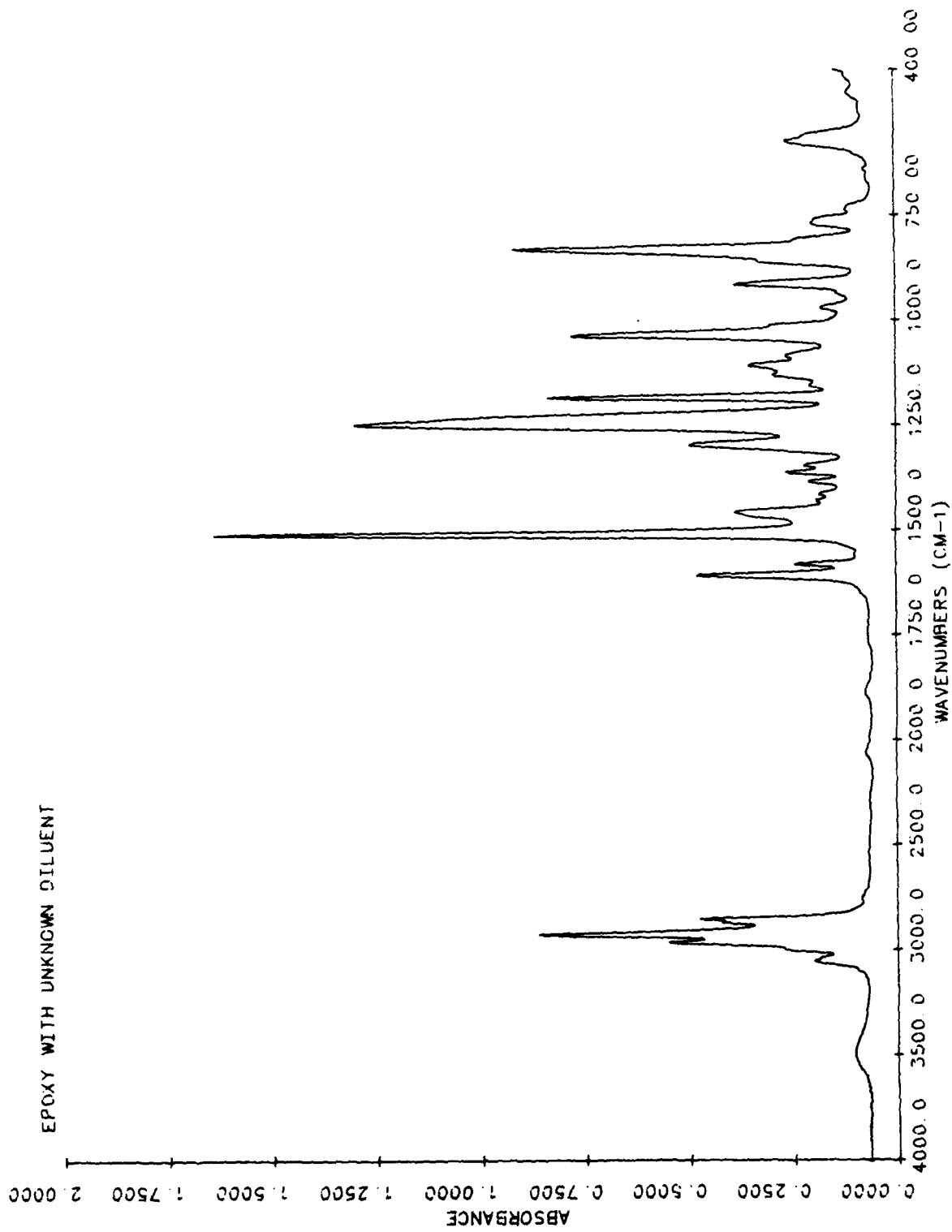


Figure 4. Spectrum of Epoxy + Diluent #1

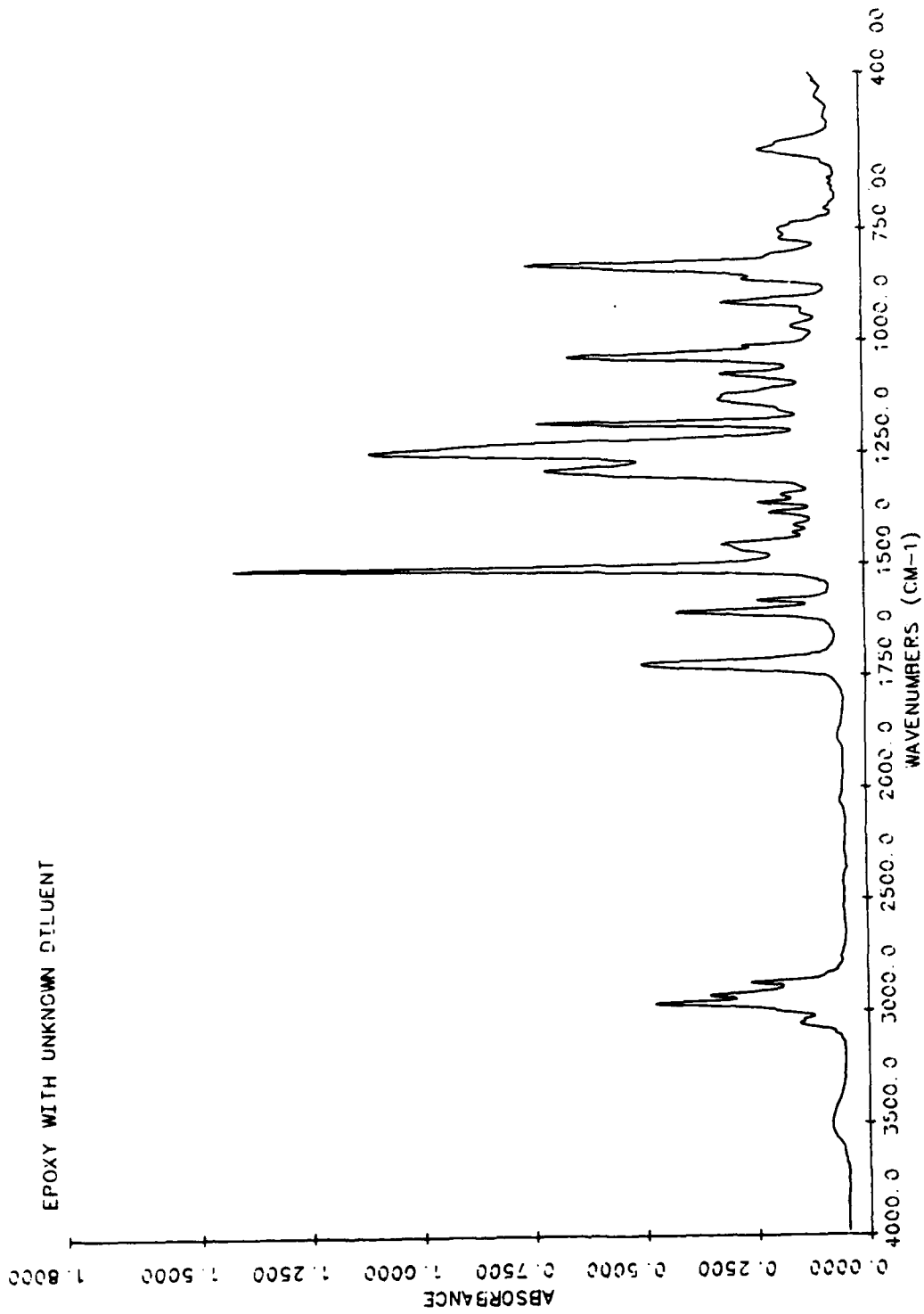


Figure 5. Spectrum of Epoxy + Diluent #2

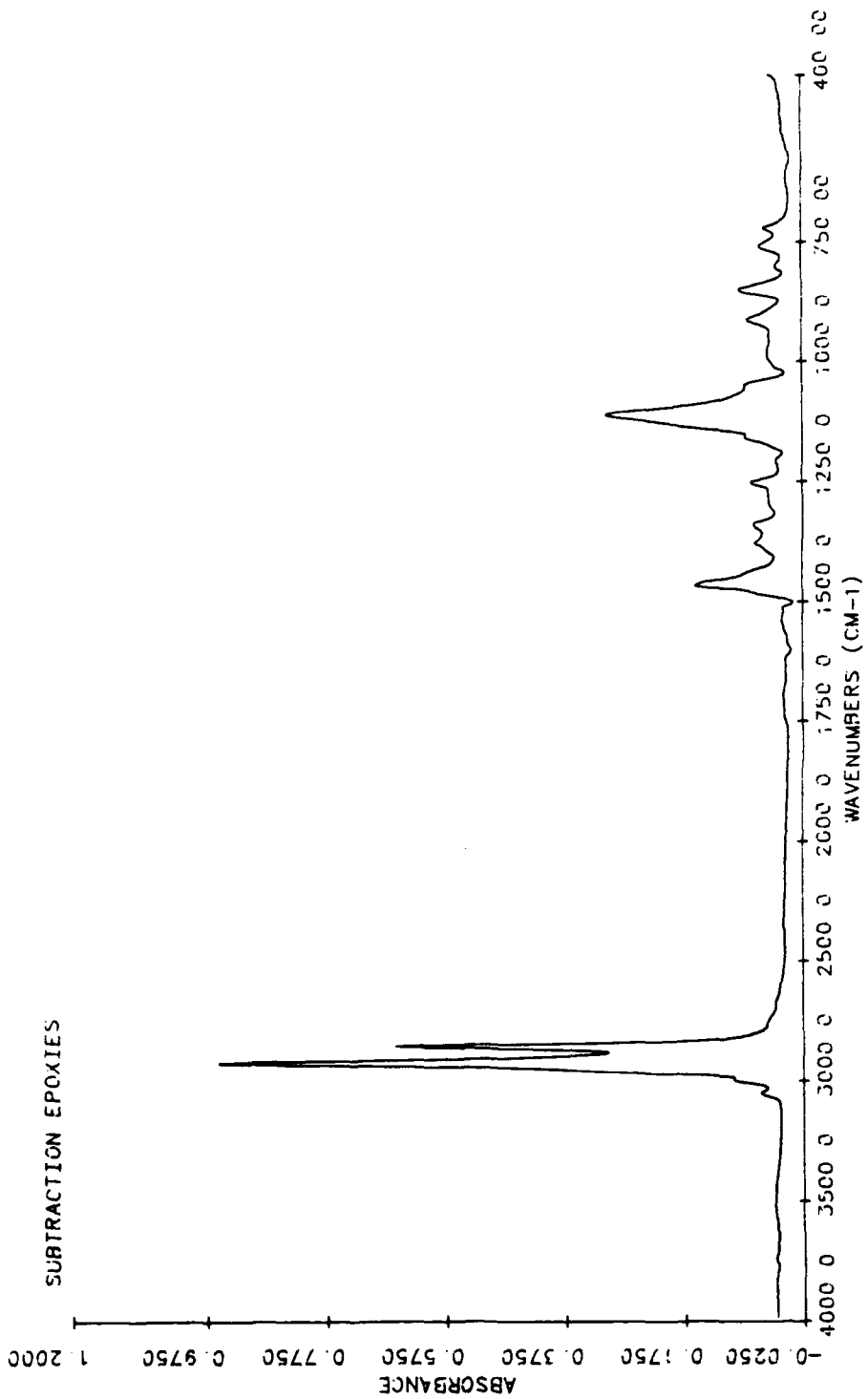


Figure 6. Difference Spectrum (Figure 4 - Figure 3)

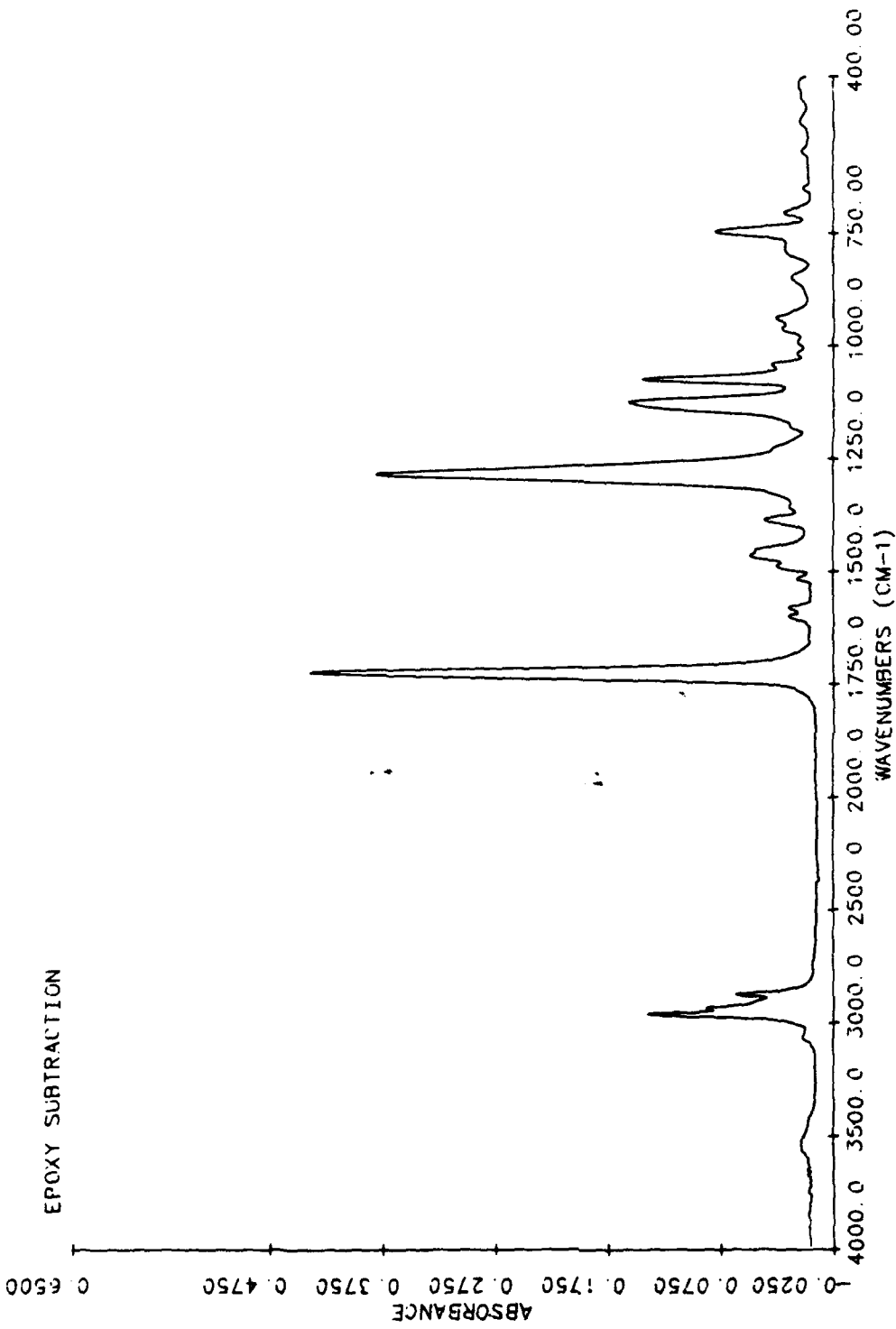


Figure 7. Difference Spectrum (Figure 5 - Figure 3)

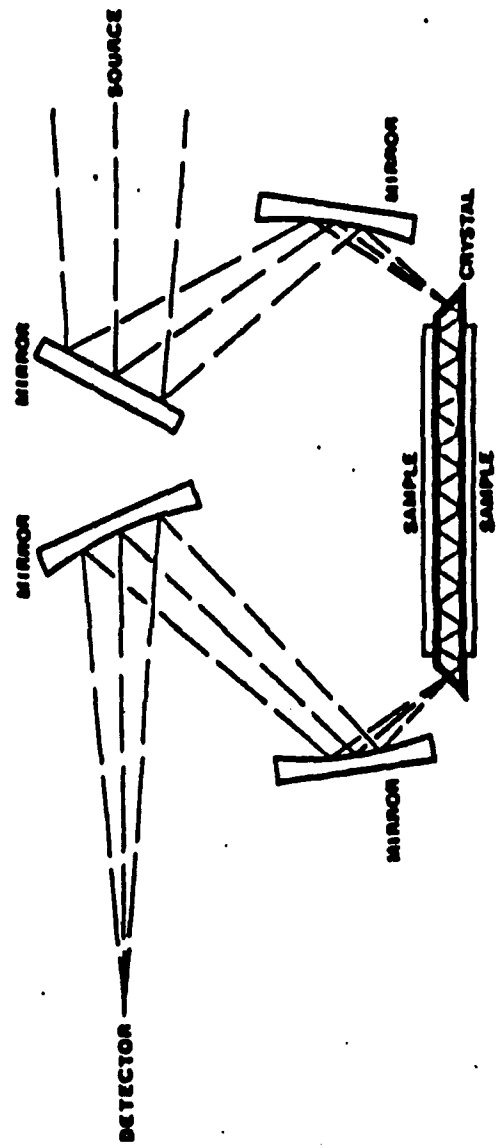


Figure 8. Schematic of ATR Set-Up

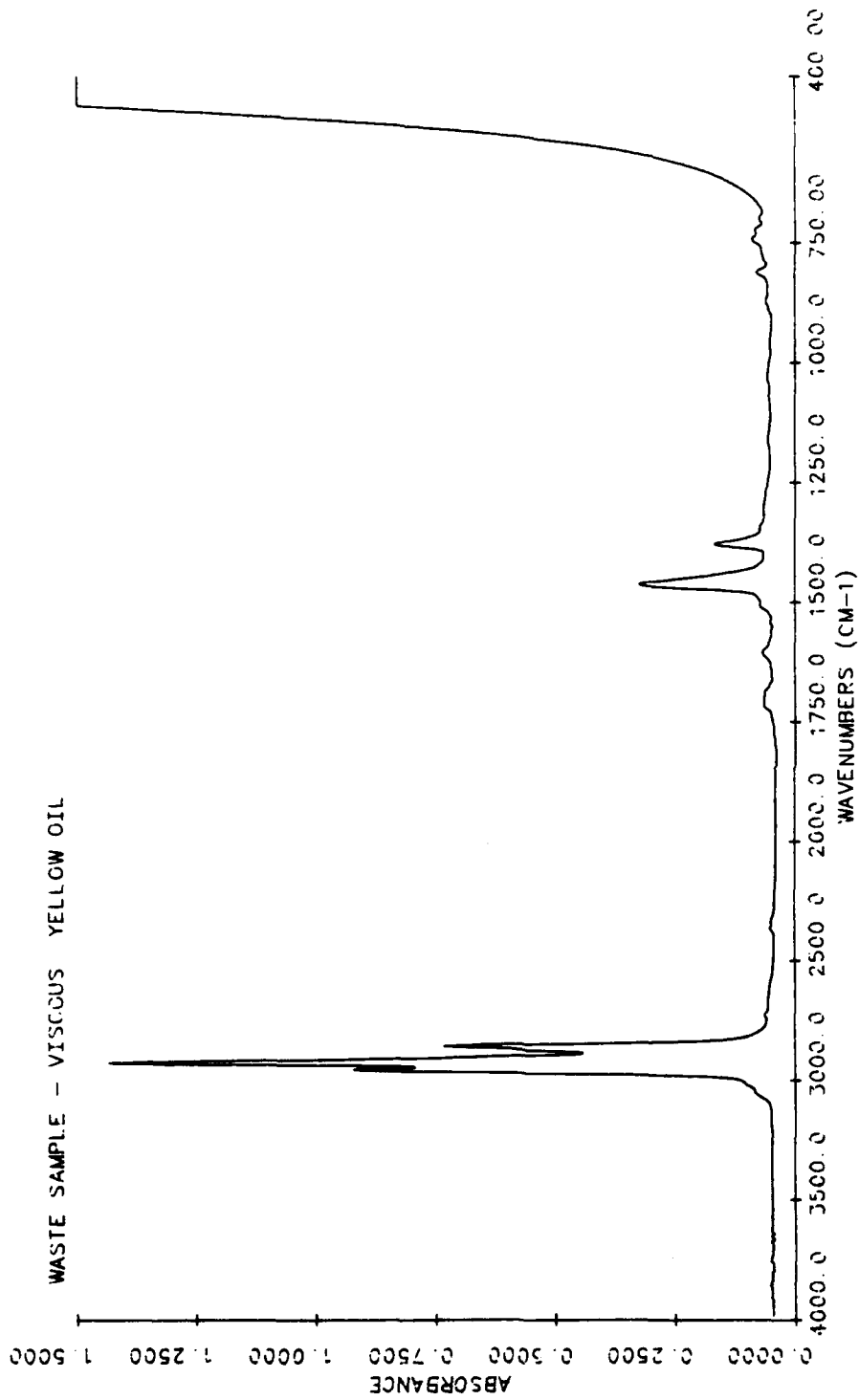


Figure 9. ATR Spectrum of Waste Oil

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