

# **Determination of Polymers and Plasticizers by Fourier Transform Infrared Spectrometer (DPT)**

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CHEM438 - 021L

*I submit this laboratory report as an original document. I assert that all ideas and discussion of data contained herein are my own work, unless otherwise referenced.*

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## ABSTRACT

After refluxing about 2.0976 g of Tygon<sup>®</sup> tubing in 30 mL of methylene chloride for an hour, the extraction was analyzed in Fourier-transform infrared (FTIR) spectrometer, and compared with the FTIR spectra of the Tygon<sup>®</sup> tubing before and after the reflux. The absorbance peak around 2,500 - 3,000  $\text{cm}^{-1}$  and 500 - 2,000  $\text{cm}^{-1}$  from the spectrum of sample before reflux disappear as observed from the spectrum of sample after reflux. It was believed that the plasticizer was removed from the sample during the reflux, and it was confirmed by analyzing the spectrum of the reflux extraction. The mass of the sample after reflux and drying was 1.2514 g, with 40.3% of plasticizer removed from the sample. The sample lost its stretchiness after reflux and was broken into tiny pieces. Unknown A, B, and C were analyzed with FTIR and were found to be polystyrene, polyethylene, and Teflon<sup>®</sup> respectively. Addition of plasticizer increased the mechanical flexibility of the polymer film, hence removing the plasticizer would cause the polymer to be not pliable.

## INTRODUCTION

For a specific molecule with different functional groups, each functional group has its own vibrational frequencies. Infrared (IR) spectroscopy can be used to characterize the frequencies and identify the functional groups in the molecule. IR spectroscopy is still widely used, and one of the uses of IR spectroscopy is in determining the compositions of polymer films.

A Fourier-transform infrared (FTIR) spectrometer is used rather than the UV-visible spectrometer due to a much higher absorbance that could be detected by FTIR. FTIR is used to essentially remove the background noise that can be seen from data collected with a UV-visible spectrometer. For FTIR, the maximum absorbance that it can acquire is around 6, which is higher than what a UV-visible spectrometer can reach, which is around 3 to 3.5 in absorbance units. Polymers have a much higher absorbance, in which FTIR is a suitable measurement to be used. For a few samples of polymers with different side chains, FTIR will distinguish these side chains by observing the difference in spectra at a particular frequency.

## PROCEDURE <sup>1</sup>

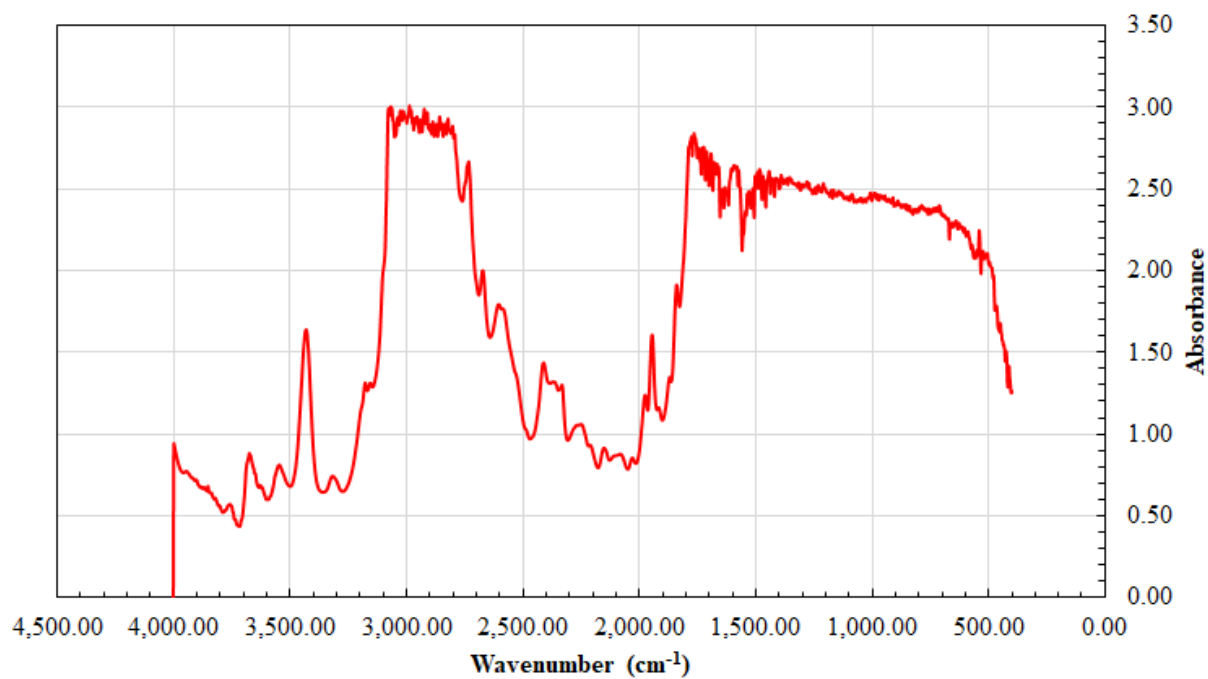
About 2 inches of Tygon<sup>®</sup> tubing in length were prepared and weighed. The weight was ensured to be about 2 g. The tubing was cut into half throughout its length to collect its FTIR spectrum. The sample was attached to a salt plate before taking its spectrum. After the spectrum was collected, the tubing was placed into a 100-mL round-bottom flask and 30 mL of methylene chloride was added into the flask. The sample was refluxed for an hour inside the hood.

While waiting for the reflux, the FTIR spectrum of three unknown samples, A, B, and C were performed. For each sample (including the Tygon<sup>®</sup> sample), the blank spectrum was collected first prior to the actual sample. The blank used was just the air atmosphere inside the instrument. The polymers of each unknown were identified by comparing their FTIR spectrum.

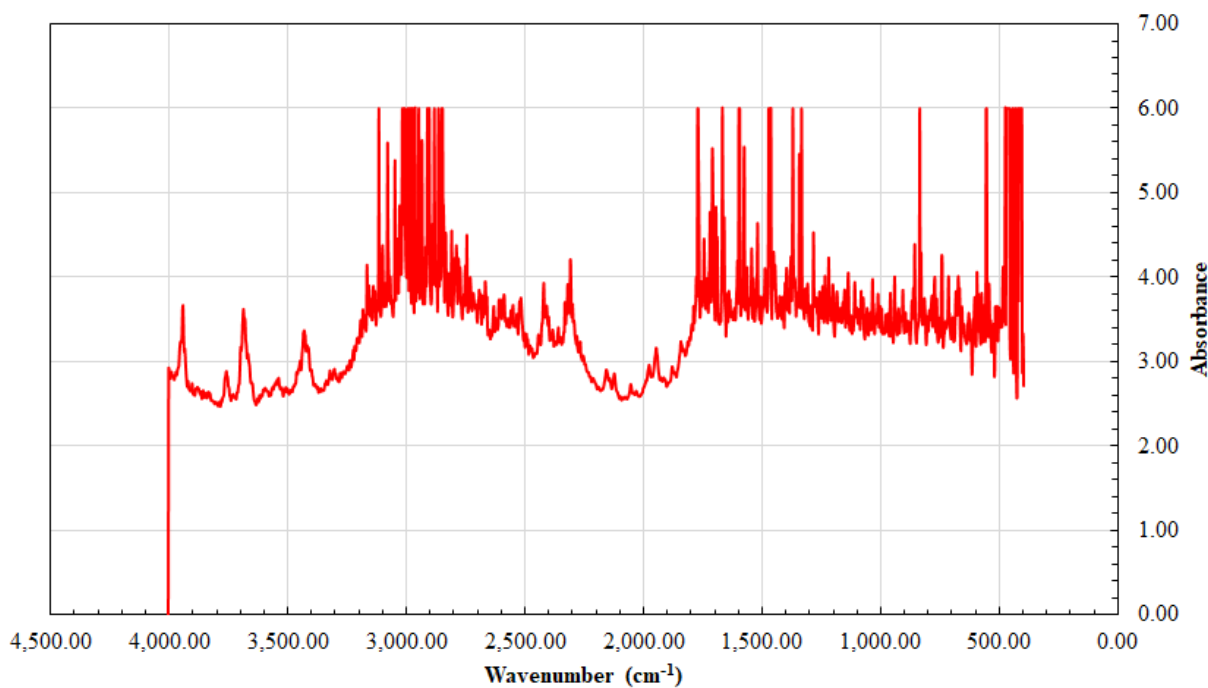
After an hour of reflux, the mixture was allowed to cool down to room temperature. The liquid was extracted into a scintillation vial. The remaining solid was left to dry before it was weighted. The FTIR of the liquid was collected, by adding the solution into the infrared (IR) cell of the instrument. The blank used was methylene chloride and its spectrum was collected first.

The dry solid was weighed and the loss in mass was calculated. The FTIR spectrum of the solid was collected by collecting the spectrum of the blank first (the air atmosphere as the blank).

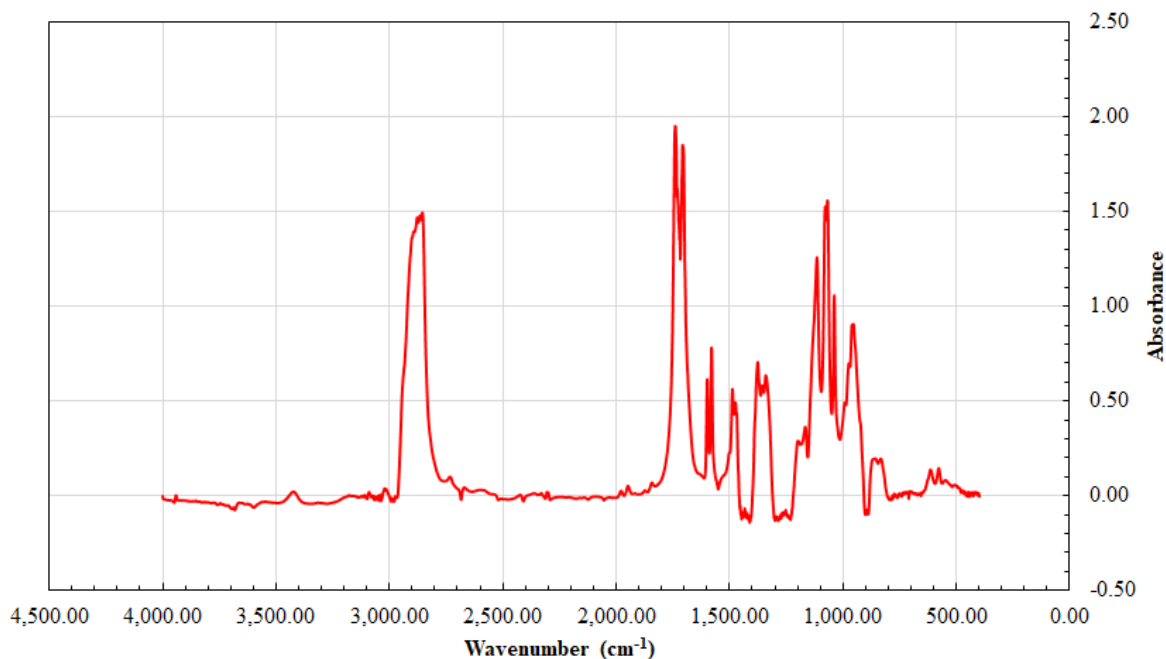
## RESULTS AND DISCUSSION



**Figure 1.** FTIR spectrum of Tygon® tubing before reflux.



**Figure 2.** FTIR spectrum of Tygon® tubing after reflux.

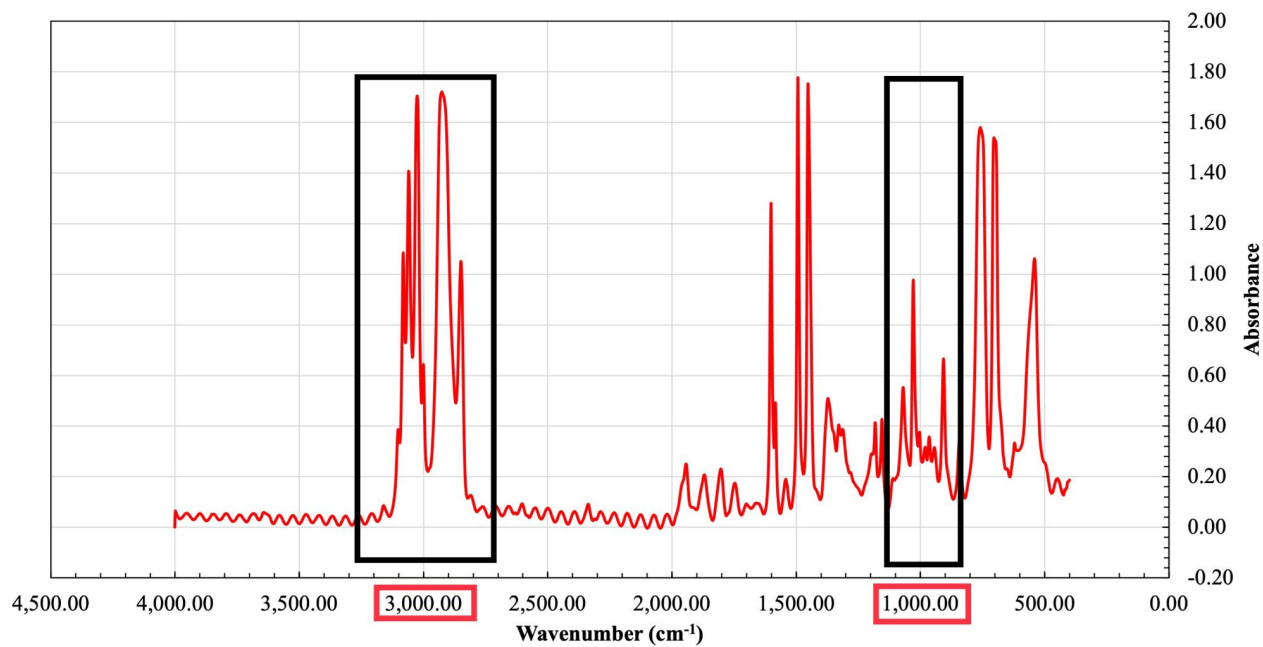


**Figure 3.** FTIR spectrum of the Tygon® tubing reflux extraction.

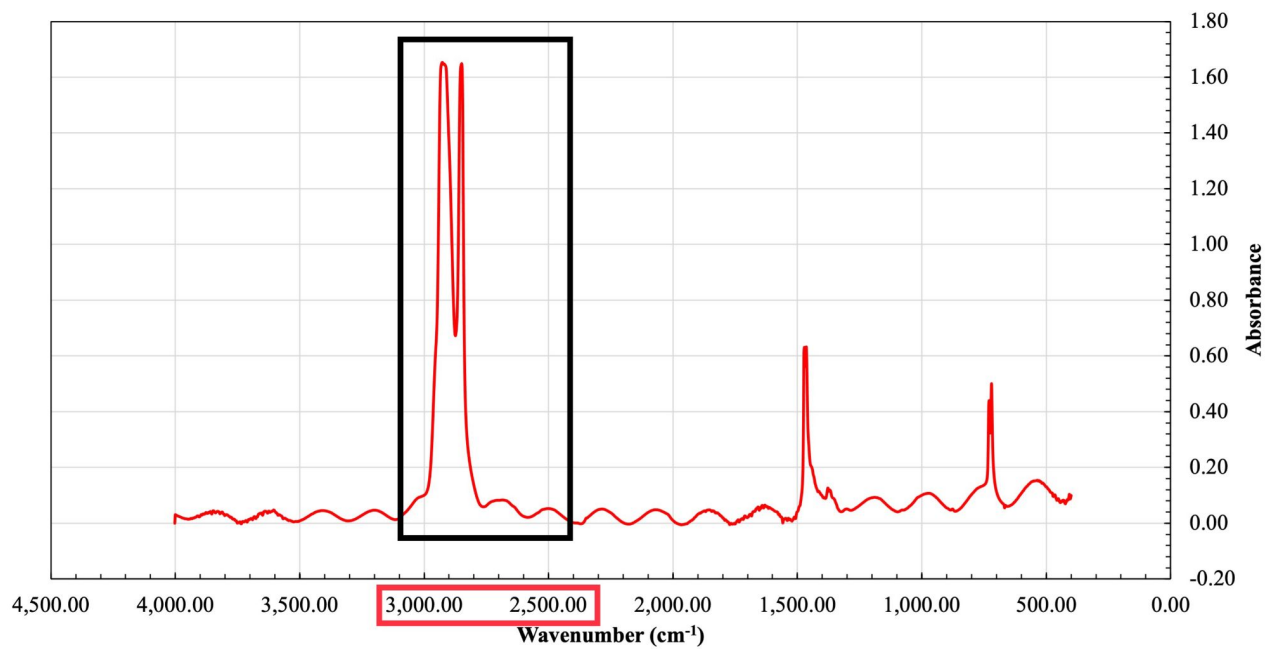
**Figure 1 - 3** depicted the FTIR spectra of Tygon® tubing before reflux, after reflux, and the reflux extraction respectively. From **Figure 1** alone, it could be seen that the absorbance peaks took place around 2,500 to 3,000  $\text{cm}^{-1}$  and within 500 to 2,000  $\text{cm}^{-1}$ . From **Figure 2**, the spectrum showed a lot of noises in absorbance data. The absorbance values measured were also greater than the spectrum of Tygon® tubing before extraction, which should not be the case. The presence of noise in the measurement could be due to the improper placement of the sample on the salt plate. During the experiment, the sample after extraction could not remain intact and was broken apart into a few pieces.

The expected results should have absorbance values of lower than 3, which should be around 1 for the maximum absorbance. This expectation was based on the fact that the plasticizer was removed and should not show any high absorbances at the wavenumber mentioned previously for **Figure 1**. For a better explanation, by observing the spectrum from **Figure 3**, the peaks could be seen occurring within 2,500 and 3,000  $\text{cm}^{-1}$  and 500 to 2,000  $\text{cm}^{-1}$ . From this extraction spectrum, the spectrum within the said range in **Figure 2** should have an approximate constant value between 0 and 1 absorbance unit.

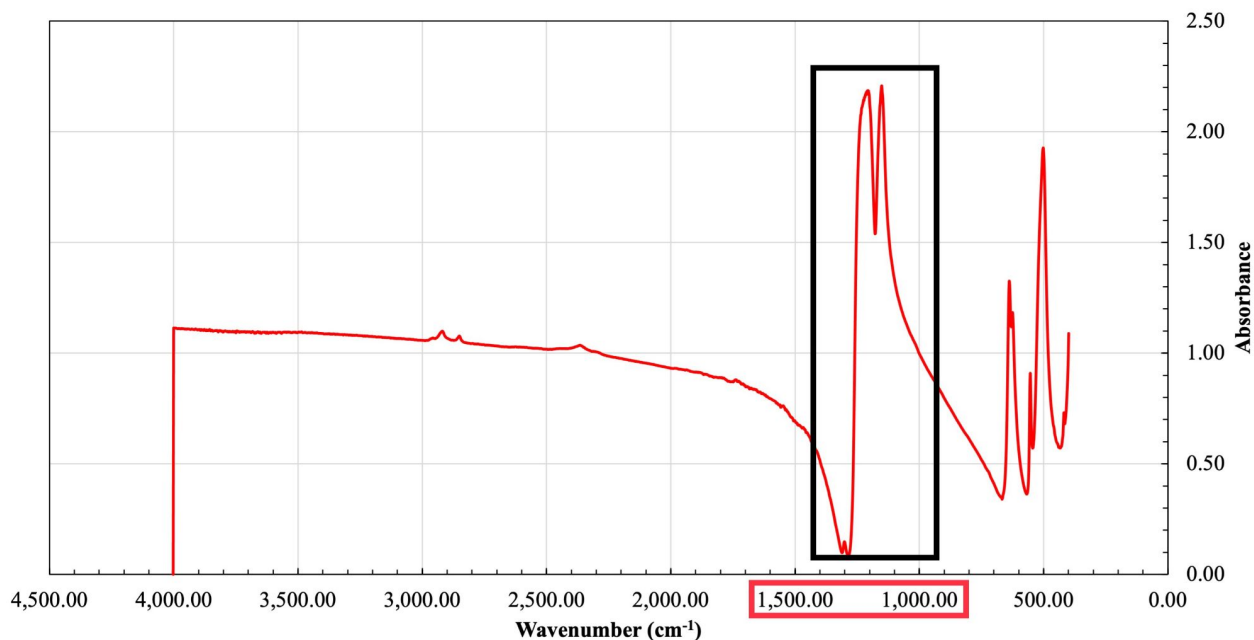
From this theory and analysis, the removal of the plasticizer from the tubing could be observed. The plasticizer could be detected from the methylene chloride extraction. The original mass of the Tygon® tubing sample was 2.0976 g. After refluxing and drying, the mass of the sample was 1.2514 g. The sample was ensured to dry completely before weighing it. Roughly, the percentage of plasticizer removed by the methylene chloride was 40.3 %. The plasticizer removal affected the pliability of the Tygon® tubing, in which the shape of the sample did not remain the same as before refluxing. The texture of the sample was hard and broken into a few tiny pieces. The plasticizer was the component that kept the original Tygon® tubing its shape and the firmness texture, as observed prior to refluxing.



**Figure 4.** FTIR spectrum of unknown A.

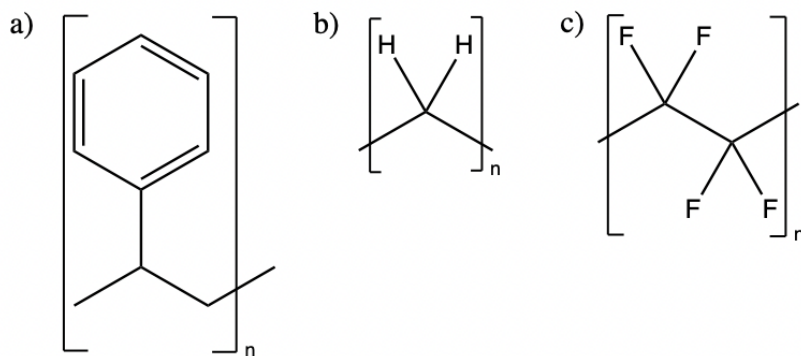


**Figure 5.** FTIR spectrum of unknown B.



**Figure 6.** FTIR spectrum of unknown C.

**Figure 4 - 6** showed the FTIR spectra of unknown A, B, and C respectively. To categorize which unknown belonged to polyethylene, polystyrene or Teflon<sup>®</sup>, the monomer of each polymer should be analyzed first.



**Figure 7.** The monomer structure of a) polystyrene, b) polyethylene, and c) Teflon<sup>®</sup>.

From **Figure 7**, the monomer styrene has a benzene ring. This corresponded to a big absorbance around 3,000  $\text{cm}^{-1}$ , alongside a big stretch at 1,000  $\text{cm}^{-1}$  and lower. Based on this, unknown A would be polystyrene. For monomer ethylene, there would be a methylene big stretch within 2,500 and 3,000  $\text{cm}^{-1}$ , which was reflected in **Figure 5**, which entailed that unknown B was polyethylene. Other than that, Teflon<sup>®</sup>, or polytetrafluoroethylene, has a C-F big stretch within 1,000 and 1,400  $\text{cm}^{-1}$ , which could be observed on **Figure 6**. Thus, unknown C was Teflon<sup>®</sup>.



The mechanical properties of each polymer could be determined by observing their monomer structure and FTIR spectrum. For the polystyrene, due to the large size of the benzene ring and having a fairly large absorbance dominating the benzene ring region ( $3,000\text{ cm}^{-1}$ ), it was expected that polystyrene was the least stretchy out of all. Both Teflon<sup>®</sup> and polyethylene have a large stretch (C-F and C-H stretch respectively), which describes the stretchiness of the polymer. Since Teflon<sup>®</sup> had a higher absorbance within  $500\text{-}1,000\text{ cm}^{-1}$  for its C-F stretch ( $2.0\text{ - }2.5$ ) than polyethylene having its C-H stretch at  $2,500\text{ - }3,000\text{ cm}^{-1}$  ( $1.4\text{ - }1.8$ ), Teflon<sup>®</sup> would be the most stretchy, followed by polyethylene.

The same observations could also be made by comparing the Young's modulus of each polymer. Young's modulus is defined as the ratio of stress below the proportional limit to the corresponding strain, where the lower the modulus, the less stiffer (more stretchy) the polymer is. <sup>2</sup> Teflon<sup>®</sup> has the lowest Young's modulus ( $0.40\text{ - }0.55\text{ GPa}$ ), followed by polyethylene ( $0.50\text{ - }1.10\text{ GPa}$ ) and polystyrene ( $1.50\text{ - }3.00\text{ GPa}$ ). <sup>3,4</sup>

The addition of plasticizer in polymer is to increase mechanical flexibility of the film. <sup>5</sup> For polystyrene itself, without introducing the plasticizer, it already has an amorphous phase with a high hardness value. With that being said, all of the films of the polymer are not pliable if their plasticizers were removed.

## CONCLUSIONS

FTIR spectrometer is a go-to technique of measurement as it has a maximum absorbance of 6, which is higher than the normal UV-Visible spectrometer, which is around 3 to 3.5. To characterize polymers as the sample, FTIR spectrometer is chosen due to the said reason, as polymers usually result in a higher absorbance. FTIR is able to differentiate the functional groups of each sample, and distinguish each polymer from one another by differentiating the side chains each polymer has.

## REFERENCES

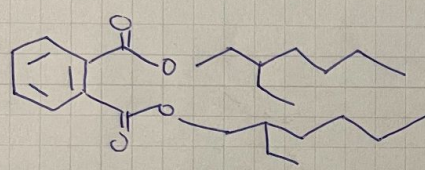
- <sup>1</sup> Cruz, F. *CHEM438 Instrumental Methods Laboratory - FTIR Determination of Polymers and Plasticizers lab*; University of Delaware: Newark, Delaware, 2018; pp 1-7.
- <sup>2</sup> Corrosionpedia. What is young's modulus? - definition from Corrosionpedia.  
<https://www.corrosionpedia.com/definition/4626/youngs-modulusr> (accessed Oct 15, 2021).
- <sup>3</sup> Dielectric Manufacturing. Material properties of teflon - polytetrafluoroethylene.  
<https://dielectricmfg.com/knowledge-base/teflon/> (accessed Oct 15, 2021).
- <sup>4</sup> Young's Modulus.  
<https://omnexus.specialchem.com/polymer-properties/properties/young-modulus> (accessed Oct 15, 2021).
- <sup>5</sup> Nasir, T.; Kim, B. J.; Hassnain, M.; Lee, S. H.; Jeong, B. J.; Choi, I. J.; Kim, Y.; Yu, H. K.; Choi, J.-Y. Plasticized Polystyrene by Addition of -Diene Based Molecules for Defect-Less CVD Graphene Transfer. *Polymers* **2020**, *12* (8), 1839.

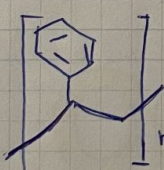
# APPENDICES

## Lab Notebook

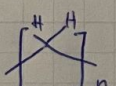
Exp. No. <u>4</u>	Experiment/Subject <u>CHEM 438 (DPT)</u>	Date <u>10/4/21</u>	15
Name <u>Abdul Fayed</u>	Lab Partner <u>Vincent Z</u>	Locker/Desk No.	

### PRE-LAB Questions

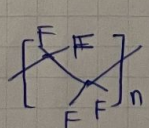
- Quantitative data is data that can be measured  $\rightarrow$  refers to numerical data. Qualitative is descriptive data (observed results). In this experiment, quantitative data will be the change in mass of the Tygon tubing. Qualitative includes the change in IR spectra after removing the plasticizers.
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Bis(2-ethylhexyl) phthalate.  
Label of peaks on NMR is attached in the PDF file.
- $\text{H}-\text{C}-\text{H}$ , wavelength  $\sim 2843-2863 \text{ cm}^{-1}$  or  $2916-2936 \text{ cm}^{-1}$   
The peaks should disappear after extraction of plasticizers.
- Polystyrene: 

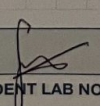
$\Rightarrow$  big stretch @ wave number  $< 1000 \text{ cm}^{-1}$   
and some stretch @ wave  $> 3000 \text{ cm}^{-1}$   
for benzene ring

Polyethylene: 

$\Rightarrow$  methylene <sup>big</sup> stretch @  $2500-3000 \text{ cm}^{-1}$

Teflon (polytetrafluoroethylene): 

$\Rightarrow$  C-F big stretch @  $1000-1400 \text{ cm}^{-1}$

Signature 	Date <u>10/4/21</u>	Witness/TA	Date
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THE HAYDEN-McNEIL STUDENT LAB NOTEBOOK

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Exp. No. <u>4</u>	Experiment/Subject <u>CHEM 438 (DPT)</u>	Date <u>10/4/21</u>	16
Name <u>Abdul Fayed</u>	Lab Partner <u>Vincent Z</u>	Locker/ Desk No.	

### Objective

The purpose of the experiment is to extract the plasticizer out of a Tygon tubing and learn the mechanical properties of the polymer through FT-IR spectrum.

### Introduction

Fourier-transform infrared spectrometer can be used to detect at higher absorbances than the UV-visible spectrometer. FTIR reached the absorbance limit of around 6. Polymers will result in high absorbance where only FT-IR can be used to analyze the structure. FT-IR helps to distinguish from one another, and it is more powerful than NMR.

### Procedure

1. Weigh the Tygon tubing before extraction and analyze in FT-IR
2. Use reflux setup for the extraction.
3. Place 2 grams of sample into 50 mL round-bottom flask.
4. Place 30 mL of MeCl<sub>2</sub> to extract the plasticizer.
5. Reflux the sample for 1 hour inside the hood.
6. Cool the MeCl<sub>2</sub> to room temperature.
7. Collect the extract into scintillation vial and analyze in FT-IR.
8. Dry the tubing after extraction then weigh it.

### Results

mass before reflux : 2.0926 g

mass after reflux and cooling : 1.2514 g  
and drying.

Texture of tygon tubing before and after : Firm before

Before: Firm, soft, intact

After: Hard, broken into pieces -

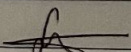
9. Note changes in the tubing based on the physical attributes.

10. Analyze unknown A, B, & C sample in FT-IR spectrum.

11. Clean up and dispose appropriately.

### Conclusion

FT-IR is used to determine the structure of polymer and differentiate from one another.

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