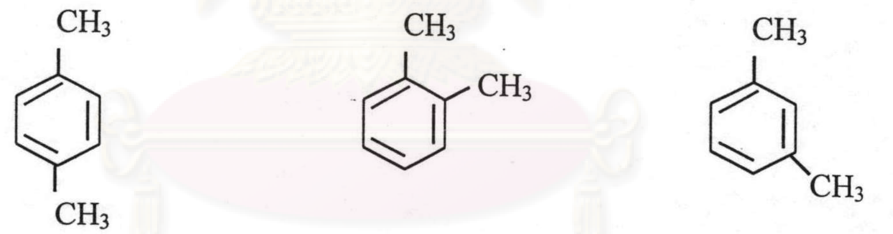
CHAPTER 1

INTRODUCTION

1.1 Overview of Xylene: Production and Use

Xylene is an aromatic hydrocarbon occurs naturally in petroleum. Most commercially available xylene is synthetically derived from petroleum and to lesser extent from coal. Three isomers of xylene are available including *ortho*-xylene, *meta*-xylene, and *para*-xylene abbreviated as *o*-, *m*-, and *p*-xylene, respectively. Mixed xylene is a mixture of the three isomers and a small amount of other components such as ethylbenzene and toluene.

Molecular formula of xylene is C_8H_{10} or it is known as $C_6H_4(CH_3)_2$. Molecular structures of the three xylene isomers are represented as follow:



p-xylene *o*- xylene *m*-xylene

Figure 1.1 Molecular structures of three isomers of xylene

In general, high purity mixed xylene is used as a solvent in chemical manufacture, agriculture, adhesives, paints, and coatings. Xylene is an ingredient in aviation fuel and gasoline. Pure isomer form of xylene is also used as a feedstock in chemical, plastic, and synthetic fiber industries. *O*-xylene is used almost exclusively for making phthalic anhydride mainly used in the manufacture of plasticizers, unsaturated polyester resins, and alkyd resins. In addition, *m*-xylene is used in the manufacture of isophthalic acid, which is used to make specialized resins. Lastly, *p*-

xylene is used mostly for making dimethyl terephthalate and terephthalic acid, which are raw materials used for the manufacture of polyethylene terephthalate (PET) used in polyester fibers, molded plastics, films, and blown beverage bottles.[1]

1.2 Quantitative Analysis of Mixed Xylene by Gas Chromatography

Quantitative analysis is applied to mixed xylene for monitoring the proportion or percentage of each isomer and other components. Typically *p*-xylene is isolated from a process stream composed of xylene isomers by selective adsorption of *p*-xylene on a solid absorbent, followed by extraction with a solvent. To monitor and control the *p*-xylene process optimally, accurate measurement of the concentrations of not only each isomer but also ethylbenzene, toluene, aliphatic hydrocarbons, and other aromatic hydrocarbons are required. The conventional analytical method used for mixed xylene is gas chromatography (GC). Each isomer of mixed xylene is separated by passing the mixture through a long narrow column of absorbent. The different gases interact differently with the absorbent material and pass through the column at different rates. As the separated gases flow out of the column, sometimes they can pass into another analytical instrument called mass spectrometer in order to determine molecular weight of the separated gas. This method requires a long analysis time (30 to 50 minutes) due to the separation of very similar hydrocarbons. Another disadvantage of GC is that the accuracy of the measurement varies since each isomer is not always baseline-separated from other peaks in the chromatogram.[2]

1.3 Quantitative Analysis of Mixed Xylene by Infrared Spectroscopy

Infrared spectroscopy is another excellent analytical method for determining quantitative information of important components in the mixture. This technique measures the intensity of infrared radiation absorbed by a sample. When a sample exposes to infrared radiation, it absorbs some of the radiation and resulting in a shift of energy state from a lower to a higher level. This is useful in regards to the identification of various compounds. The advantages of spectroscopy analysis include: [3]

- Direct more true analysis of component,
- No toxic reagents are required for analysis,
- Rapid results are obtained, once calibrated,
- Suitable for use at-, on-, or in-time in processing plants, and
- Simultaneous analysis of several components is possible.

Measuring the concentration of an absorbing species in sample by infrared spectroscopy is accomplished through application of Beer-Lambert law. The Beer-Lambert law is the linear relationship between the absorbance and concentration of the sample by the following expression:

$$A = abc \qquad [1.1]$$

where A is the absorbance of the sample, a is the absorptivity, b is the pathlength of the sample, and c is the concentration of the sample.[4]

This assumption is the key step of quantitative infrared analysis. The bandshape of an infrared spectrum depends on the type of sample and the absorbance of the sample is directly proportional to the concentration of the sample.

1.3.1 Problem in Quantitative Infrared Analysis of Mixed Xylene

Quantitative analysis of mixed xylene from xylene spectrum to qualify three xylene isomers that consisting in mixed xylene must rely on the out-of-plane C-H bending vibrational region (600 to 850 cm^{-1}). The bands used for the analysis are:

o-xylene	739 cm^{-1}
m-xylene	768 (or 691) cm^{-1}
p-xylene	795 cm^{-1}

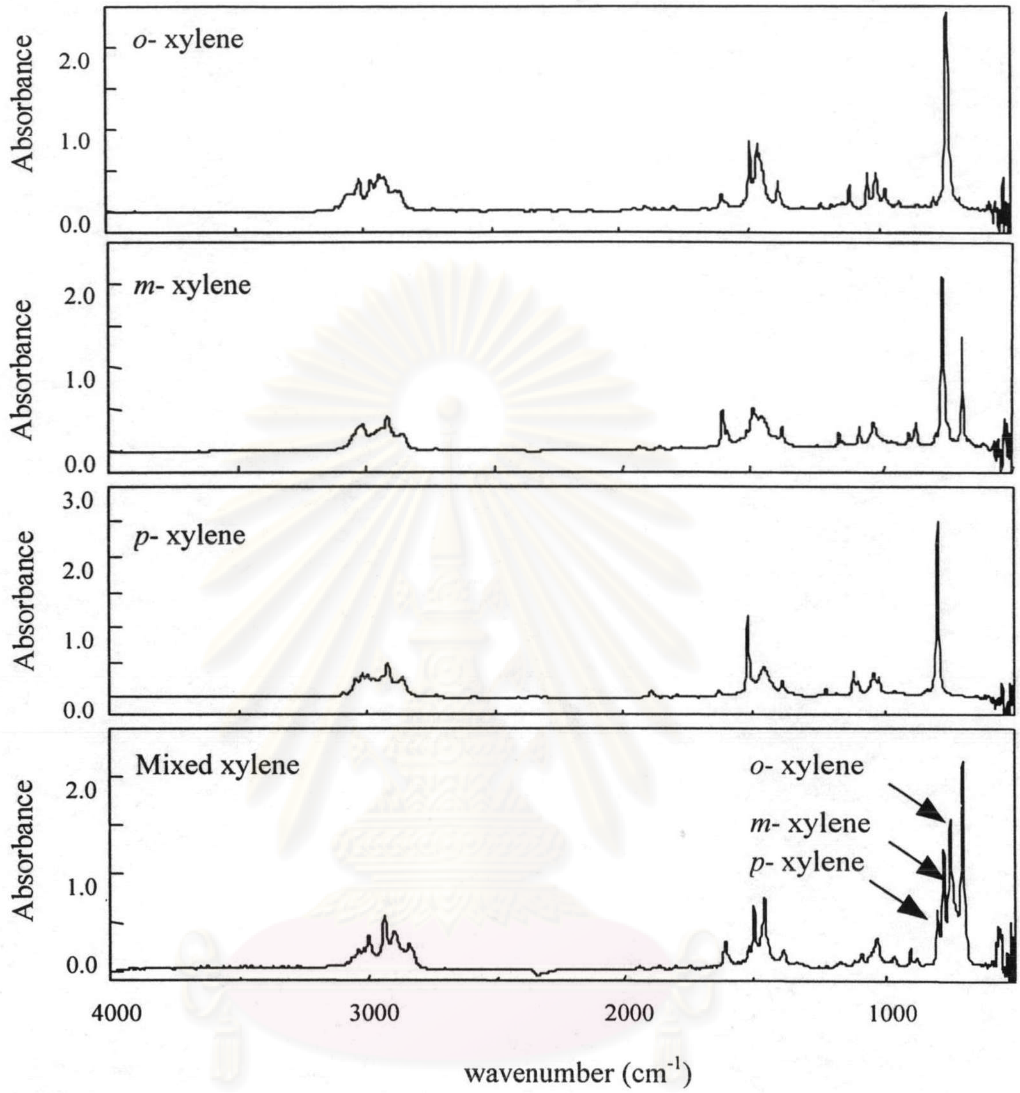


Figure 1.2 Infrared spectra of three isomers of xylene and their mixture.

This conventional method requires an isolated infrared absorption band. But the spectral features of the three xylene isomers are very similar over the infrared region due to the structural similarity. This region presents the peak overlap that exhibits the deviation from Beer-Lambert law. In an actual experiment the spectra may be detected under condition of high level of noise, the spectral information obtained is not correct and thus reduces the quantitative accuracy.

1.4 Application of Mathematical Algorithm For Solving the Problem in Quantitative Infrared Analysis of Mixed Xylene

Chemometrics is the utilization of mathematical and statistical methods for handling, interpreting, and predicting chemical data especially when analyzing multicomponent mixture. The several techniques of chemometrics are used for the quantitative analysis of multicomponent mixture such as least-squares and cross-correlation.[5] Principle component factor analysis or well known as factor analysis (FA) is a powerful technique of chemometrics. It has been successfully applied in a number of areas of analytical chemistry. In quantitative spectroscopy, this technique has the advantages that it can be applied under various conditions such as: [6]

- Complete spectral overlap,
- Signal-to-noise ratio less than one, and
- Failure of the Beer-Lambert relation.

1.5 The Objective of This Research

According to advantages of factor analysis and infrared spectroscopy, these techniques will be employed for the quantitative analysis of mixed xylene in order to determine the concentration of each isomer of mixed xylene.

1.6 Scope of This Research

1. Study the ability of factor analysis for the quantitative analysis of mixed xylene by infrared spectroscopy.
2. Demonstrate technical feasibility of the quantitative analysis of mixed xylene by infrared spectroscopy and factor analysis in comparison to conventional technique such as gas chromatography.