

CHAPTER I

INTRODUCTION

1.1 Sample handling for liquid and solid samples in transmission technique

Infrared (IR) spectroscopy is one of the most ubiquitous techniques used in the introductory organic chemistry laboratory. Cells prepared from alkali-halide such as sodium chloride (NaCl), potassium bromide (KBr) and potassium chloride (KCl) are widely used due to their transparent properties in the IR region. A common problem with those cells is that it adsorbs moisture and become fogged. Polishing is required to store the cell to a more transparent state.

Liquid may be analyzed in their neat form by placing a small amount of sample on a NaCl plate and then placing a second plate on top to form a sample film. The plates are then placed in an appropriate holder in the sample compartment of the instrument. This technique provides adequate spectra for qualitative use.

Solutions of solid or solid materials can also be analyzed by IR spectroscopy. Solvents should be chosen that do not have absorbances in the region of interest. Influence of solvent on the absorbance of the solute should be considered. In practice, it is desirable to analyze neat materials for qualitative analysis.

A method commonly used for analysis of solid samples is the mull technique. The technique consists of grinding the material into a fine powder and then dispersing it into a liquid or solid matrix to form a mull. Liquid mull has been by combining the powdered analyte with Nujol (heavy hydrocarbon oil). The disadvantage of Nujol is that hydrocarbon bands may interfere with analyte absorbances. A second method involves grinding the powdered analyte with dry KBr and forming a disk. Care must be taken when preparing the disk to protect it from moisture. It is very common to see absorbances for moisture when using KBr disks. Another technique for handling samples is the use of disposable infrared cards. This technique may be used when a limited amount of analyte materials is available.

1.2 Disposable IR card

New IR card has been developed to offer a low-cost sampling alternative to conventional methods such as attenuated total reflectance (ATR) analysis or liquid transmission cell analysis. The IR cards were manufactured to use for qualitative IR transmission analysis of organic liquids, materials soluble in organic solvents, semi-solids and pastes.

The commercially available cards (see Figure 1.1) contain an IR transparent materials (the alkali-halide support substrates such as KBr, KCl and NaCl) were used in IR cards. These materials transmit enough energy for spectroscopic and other types of IR analysis, are sandwiched between cardboard frames that have centrally located clear apertures filled by the crystal window. The beam of the instrument can be directed through this aperture and the window is held in place by pressure sensitive adhesive that coats the interior of the cardboard frames. In addition, the polymer substrates have uncomplicated absorption peaks were also employed as sample substrates. Both polymer substrates are polyethylene (PE) and polytetrafluoroethylene (PTFE). The PTFE cards are highly effective in the $4000\text{-}1300\text{ cm}^{-1}$ range and PE cards are well suited for substance requiring interpretation in the fingerprint region ($1600\text{-}400\text{ cm}^{-1}$). The commercial IR cards are universally sized to fit major FT-IR spectrophotometers without the need for additional apparatus or special alignment features. A small amount of sample is applied directly onto the card substrate window appearing through the aperture of the card. This can be accomplished with using something smooth or rounded that will not damage the substrate. The IR card is then placed into the sample compartment to the spectrophotometer and scanned.



Figure 1.1 Commercial IR cards

Advantages of the home-made IR cards are reducing sample preparation time, disposable card and improving productivity. In addition, these cards provide a fast and clean method to run IR spectra.

1.3 The objective of this research

In this research, the IR card from PTFE film was set off to make similar commercial cards using Teflon tape available in most hardware stores for very cheap price per roll. Therefore, a lot of home-made IR cards can be made from only a roll of Teflon tape. The objective of this research is to create the IR card from thin PTFE film for various types of sample analysis (solids, viscous liquids, volatile liquids, thin-films and gases). And develop surface of PTFE film with coating gold (Au) particles, it is expected to have a high efficiency for thin-film or low-concentration sample analysis. The experimental investigations of home-made IR card will be performed and will be explored their limitation, advantage and disadvantages.

1.4 Scope of this research

1. To create the new home-made IR card from thin PTFE film by stretching the PTFE film.
2. To study the sensitivity of thin PTFE film for sampling samples.
3. To investigate the efficiency of the home-made IR card for analyzing of solids, viscous liquids, volatile liquids, thin-films and gases.
4. To coat Au onto the surface of PTFE film in order to enhance the sensitivity of PTFE film for low-concentration sample analysis.