application note

# Sample Preparation for IR Spectra - Liquids & Solids

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Bio-Rad's laboratories have prepared and analyzed thousands of samples by means of infrared spectroscopy. These samples range from commercial products like polymer beads and liquid surfactants to high purity organic compounds suitable for their standards collection. A wide variety of techniques have been developed by spectroscopists and spectral technicians in order to obtain high-quality infrared spectra from these diverse materials. The purpose of this Application Note is to communicate Bio-Rad's knowledge of infrared sample preparation to you. In this Application Note, selection of technique based on physical characteristics of the sample will be discussed.

The information desired from a spectrum largely determines how the compound is to be prepared for examination. A compound in the crystalline state generally yields a spectrum which has more absorption bands than the spectrum of the same compound when examined in the liquid state or in solvent solution. The absorption bands in the spectrum of a solid compound, examined in the crystalline state, or a liquid compound, examined in the pure concentrated form (neat), are found, usually, at lower frequencies than the absorption bands in the spectrum of the same compound when it is examined in the vapor state or in solvent solution. The degree of absorption band shifts depends upon the presence or absence of intermolecular and intramolecular forces.

## Liquids:

Liquids are prepared by placing a small amount of sample between two infrared transparent windows. The crystals are pressed together to form a thin layer of the sample. The formulation of the sample determines which window material is used. For non-aqueous samples, the window material is KBr. For aqueous samples, KRS-5 is preferred.

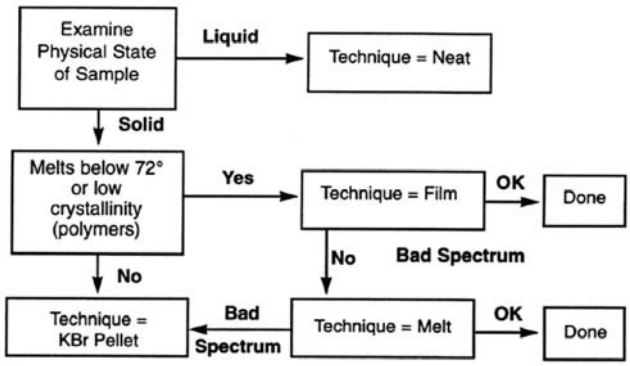
## Solids:

Solids present a variety of challenges to the spectroscopist. The melting point of the sample often points us to which technique to attempt first.

- For samples with a melting point less than 72° C, filming on a KBr window from a suitable solvent is initially attempted. If this is not successful due to a poor baseline or insolubility, a melt between two KBr windows is attempted. If this is not successful, the sample may be attempted as a KBr pellet.

- For samples with a melting point greater than 72° C, the technique of first choice is a KBr pellet. For polymer samples, filming the sample is attempted first, followed by a melt and then KBr pellet techniques.

- For samples with an unknown melting point, an examination of the crystallinity of the sample can indicate which technique may be successful. Highly crystalline samples tend to yield good KBr pellets. For samples that exhibit low crystallinity, films and melts tend to give better spectra.

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