

CH424 Instrumental Analysis

Sample handling technique for IR Analysis

1. Liquid Samples

Liquid samples can be run neat or by dissolving in a solvent. The sample concentration and path length should be selected to obtain the transmittance in the range of 15%-70% in order to get a good IR spectra. This will correspond to about 0.02 mm cell thick in the case of most neat liquids, and concentration of 10% and cell length of 0.1 mm in the case of most solutions. The solvent selected must be transparent in the region of interest. Neat liquids can be analyzed between salt plates made of NaCl or KBr.

Non- or low volatility liquids can be analyzed by placing a drop of the sample onto specially prepared thin polyethylene (or other) polymer substrates. These supports (called "IR cards") are cheap and disposable. They absorb IR only in well-known, narrow bands, which depend on the material. These absorption can be accounted for using the clean substrate as a background. The absorptions are ratioed out in the final step of sample spectrum generation.

2. Solid Samples

Solid samples can be prepared for IR analysis by several methods:

Mull technique

The mull technique is probably one of the most common and easiest ways to prepare a solid sample. The sample is prepared by first grinding about 5 mg of a solid sample in a mortar, then adding 2 drops of the mulling oil (Nujol or Fluorolube mulls) to the sample, and grinding the mixture thoroughly. It is important that the suspended particles obtained must be less than 2μ in order to avoid excessive scattering of radiation. Then, paste the suspended particles on one of the IR salt plates, and cover with the matching salt plate.

Pellet technique

This technique is based on the fact that a sample in KBr powder can be compressed under pressure with or without vacuum to form transparent disks. In this technique, a solid sample of approximately 2-3 mg is allowed to mix with about 0.5- 1 g of KBr (which is transparent to IR). Thoroughly grind the mixture in a mortar, then press the mixture in a pellet die under a pressure of about 6000-10000 psi to obtain a transparent disk. Good dispersion of the sample in KBr is critical. It should be pointed out that bands near 3448 and 1639 cm^{-1} from moisture often appear in the spectra obtained by this technique. One should avoid moisture by, for example, freeze-drying of the sample as needed.

KBr disk can be prepared easily with a Mini-Press accessory. The KBr sample mixture is placed in the nut portion of the accessory with one bolt in place. The second bolt is then introduced, and pressure can be applied manually by hand-tightening the bolts. A pellet of a sample is obtained after removing of the bolts at both ends, that leaves the pellet in the nut that now serve as a cell which can be attached to a cell holder directly.

There are a number of other sampling techniques specific to the type or state of the sample. Special cells have been developed for semi-transparent liquids and weakly absorbing gases. Pastes and other semi-solids are routinely analyzed with the help of an attenuated total reflectance (ATR) attachment. Monolayers on reflective metal surfaces can be measured using an external reflectance accessory. The specific application of FT-IR to various samples yields quite a number of methods.