application note

Sample Preparation for IR Spectra - Film Technique

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In Bio-Rad's laboratories, the film technique is used for samples with melting points below 72°C and for samples with low crystallinity, such as polymers. The film technique is also attempted when other methods fail to produce a good spectrum.

General Procedure for Casting a Film

The sample is first dissolved in a suitable solvent. Several drops of the solution are then placed on an inert substrate and the solvent is evaporated off leaving behind a thin film of the sample on the substrate. The film is then either peeled off and placed in the spectrometer; or, if the substrate is transparent in the infrared, the substrate with the sample can be placed into the spectrometer.

Selecting a Suitable Solvent

The main criterion for a solvent (besides the obvious one, dissolving the sample) is that it should be easy to remove. This implies a low boiling solvent. The less heat applied to evaporate the solvent, the less the sample will be affected. In addition, the easier it is to remove the solvent, the less solvent will be left behind.

In our laboratories, the following list of solvents are attempted first: chloroform (BP. 61.2°C), acetone (BP. 56.2°C), trichloroethanol (BP. 151°C), o-dichlorobenzene (BP. 180.5°C), and water (BP. 100°C). These five solvents are suitable for about 85% of the samples for which the film technique is attempted. The spectra of the pure solvents should also be kept on hand as a reference.

Comparison of the solvent's spectrum to the cast film is considered mandatory to determine if any solvent was left behind. It is also a good practice to generate a new reference spectrum each time a solvent is reordered.

Selecting the Substrate

In Bio-Rad's laboratories, no attempt is made to peel the film off the substrate. The substrate is placed with the film into the spectrometer.

Therefore a sub-strate which is transparent in the infrared is needed. The most common choice is a KBr crystal unless the solvent is water, in which case a KRS-5 crystal is used. If the decision is made to peel off the film, then glass is generally a good choice.

Casting the Film

It is our experience that it is best to use a small amount of a dilute solution (3-5 drops) and cast several thin films on top of each other, rather than to produce one thick film from a concentrated solution or from using a large amount of the solution. This minimizes the possibility of trapping any solvent within the film.

Sometimes, when casting films of crystalline materials, a spectrum may show a significant amount of scattering and give a poor sloping baseline. This problem is more common when trying to cast a single thick film, but also occurs when casting several thin films on top of each other. This is most like a result of the first crystals precipitating out and acting as "seed" crystals to produce larger crystals. It is these large crystals which cause the light to be scattered, hence, a poor baseline. To avoid this problem in our laboratories, we will often cast a thin film on both surfaces of the crystal, and occasionally on both surfaces of two crystals, resulting in a total of four thin films. This technique corrects most scattering problems.

When evaporating off the solvent, keep the solution moving over the crystal. This will help you get a film of even thickness. We usually place the crystal on a small disposable cardboard card (about 2" x 3") and either rock the card back and forth to keep the solution moving, or use the end of the pipet to smear the solution over the crystal. If heat is required to remove the solvent and the crystal is a hydroscopic material such as KBr, then you should first heat the card to drive off any residual moisture in the card. If you do not do this, the bottom surface of the crystal will absorb this moisture and cause it to become cloudy, which can cause a sloping baseline in your spectrum.

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In our laboratories, we use a heat lamp to gently evaporate off the solvent. This should always be done in a fume hood to remove the vapors from the lab environment. It is important to observe all necessary precautions, especially when working with flammable solvents.

Potential Problems

The two most serious problems that can occur with the film technique are an uneven film or residual solvent in the spectrum. The problem with an uneven film is spectral non-linearity. The problem of a residual solvent must be vigilantly searched for whenever the film technique is used. Always check the resulting spectrum for the major bands of the solvent. If it appears that some solvent remains in the spectrum, it is sometimes possible to drive off the last of the solvent by applying more heat.

If you are still unsure whether the sample or the solvent caused a particular band, then the sample must be re-run using a different technique or a different solvent which does not contain that band.

Another possible problem is that some samples may oxidize easily in the presence of heat and oxygen. This often results in a small band in the C=O region around 1740 cm^{-1}.

There are several ways to prevent or minimize this oxidation. Evaporating off the solvent in an inert atmosphere such as nitrogen, reduces the availability of oxygen. Applying less heat will also minimize the problem. You can use less heat by changing to a lower boiling solvent if possible, or by removing the solvent in a vacuum oven.

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