

One common technique for preparing a sample for FTIR analysis is to mix the finely ground sample with powdered spectroscopic grade KBr and press a pellet from the mixture. The pellet may then be analyzed using transmission spectroscopy. Although this process may seem straightforward, certain complications do exist.

Obviously, this technique is not suitable for liquids. For non-powder solid samples, the sample must first be ground into a powder, prior to mixing it with KBr. For reproducible results, the powder size of the sample and of the KBr should be kept uniform. Also for reproducible results, efficient mixing with KBr must be accomplished, something which requires considerable technique considering that the ratio of KBr to sample is typically 100:1. During grinding and mixing procedures, the sample and KBr should be kept dry, to avoid water band interference in the spectra. On humid days, it may be necessary to dry the mixture in an oven prior to making a pellet.

Prior to placing the sample in the press, the analyst must be sure that the press itself is dry. Heating of the press, or sections thereof, may be required to remove water. The sample is then placed in the press. Some mechanical assembly is typically required. With some presses, the quality of the pellet produced depends on a careful amount of mixed sample being placed in the press. Pressure is then placed on the sample to produce the pellet. During this procedure, vacuum is often applied to the press. One must also know the correct amount of pressure to apply to produce a good pellet. The sample is then allowed to stand for a few minutes and the press is disassembled.

Some presses allow the pellet to be analyzed directly without removing it completely from the press body. With others, removal is required. Here, breakage of the pellet is a possibility. In both cases, special transmission sample holders are required. Here again, the independent pellet is subject to breakage when handled. Note that a second KBr pellet, one without sample, must be run as the reference. This same pellet, if kept dry, can be used as the reference for a number of different sample pellets.

The above process involves a considerable amount of experience and technique to repeatedly produce usable pellets. The time involved in gaining this expertise, as well as the actual sample preparation time, are serious drawbacks to the KBr pellet technique.

One alternative to KBr pellets is the use of ATR. Here, sample preparation is minimized. Solid and powder samples may be analyzed directly. No mixing of samples is required, as the built-in small pathlength of the ATR technique provides sufficient attenuation for virtually all samples. Air is run as the reference sample. The Harrick Scientific [MVP-Pro™](#), using a Si ATR element, the [SplitPea™](#), using a silicon ATR element, and the [Meridian™](#), using a diamond ATR element, are three examples of single reflection ATR accessories which can be used to replace the KBR pellet technique and associated equipment.

A second alternative to KBr pellets is the use of diffuse reflection. As with ATR, both solids and powders may be analyzed. With solids, however, the surface must be rough or be roughened. Although many samples may be analyzed neat, it is often necessary to dilute the sample by mixing with KBr to reduce distortions due to reststrahlen bands. The Harrick Scientific [Praying Mantis™](#) and the [Cricket™](#) are two examples of diffuse reflection accessories that can also be used to replace KBR pellets.