

How to Prepare Samples for IR Spectroscopy – KBr pellets

Reading: Mohrig, et al., *Laboratory Techniques in Organic Chemistry*, 4th edition, Chapter 21, Section 21.5.

KBr pellet press (for solids only).

1. For *solid* compounds, grind ~1-2 mg of the unknown in the special mortar and pestle (both made of agate, expensive!) until it is shiny (the goal is to grind the sample to a particle size of <2 μm , which takes at least 2 minutes). Then add ~80 mg of anhydrous KBr and mix quickly with the sample (since KBr will absorb moisture from the air, therefore always keep the lid of bottle of KBr tightly sealed since it quickly absorbs moisture).
2. Add ~half of the contents (~30-40 mg) of the ground mixture to the press with one of the bolts screwed *halfway* in the press. Screw in the other bolt and finger tighten. Using the correct wrench and the vise, slowly tighten the bolts to approx 20 ft-lbs of torque. Be sure that the wrench is used correctly to avoid stripping the bolt head. Leave the sample under pressure for 30 seconds before carefully removing one of the bolts. A successfully formed pellet is almost translucent [not cloudy--if it's a little cloudy the pellet will still work although the resolution will be less (peaks will be broad)].
3. Obtain the IR spectrum by carefully placing the KBr press in the holder in the sample chamber of the IR. (see IR instructions for use)
4. Poke out the KBr pellet with the eraser of a pencil. Do not use a spatula or scratch the press or bolt, since it might damage the smooth surface. Clean the mortar and pestle with distilled water, then finally acetone (back and forth until all of the inorganic (KBr) and organic materials have been removed), and dry with a tissue inside and out and then return to the oven to dry. (Both acetone and water will adversely affect the quality of your IR spectrum, so the KBr press must be completely dry and solvent-free before use.)

Using the QuickPress[®] KBr pellet maker

1. Perform **Step 1** as above for the MiniPress.
2. Add ~30 mg of the prepared sample to the special die (see instructor for directions). Small anvil, large anvil, plus center ring.
3. Place die (small anvil down) in the QuickPress and close the handle until it "clicks" closed. Adjust setting on the end of the QuickPress so that when the handle is depressed it is difficult to close.
4. Clamp the handle and apply strong even pressure for ~30 seconds, then open the QuickPress and carefully remove the die. (Hold hand over the opening of the die so that it doesn't fall out)
5. Remove both ends of the die and place the central part of the die in the Spectrometer.
6. Take spectrum as instructed.
7. Clean the die by using the longer anvil of the die to poke out pellet, then clean with a dry Kimwipe.
8. Completely clean if needed with DI water, then acetone. Wipe dry with a Kimwipe.