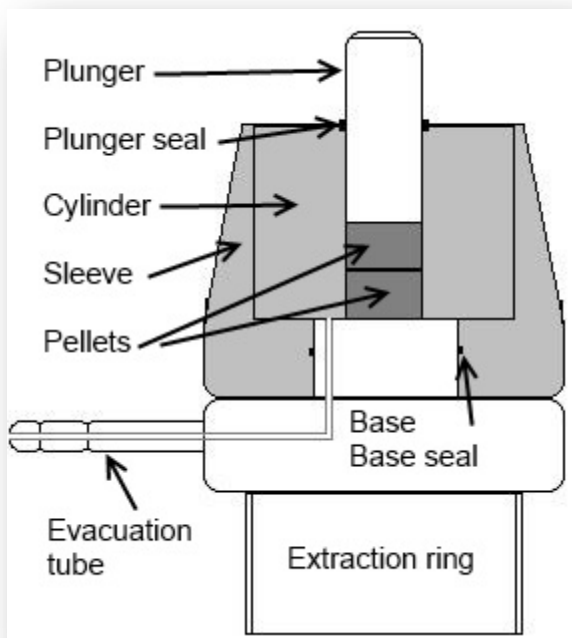


5 mm – 16 mm evacuable KBr dies

- Instructions for use
- Dies with water jacket
- Type of steel used for pellets
- KBr – general information and disc preparation for IR spectrophotometry

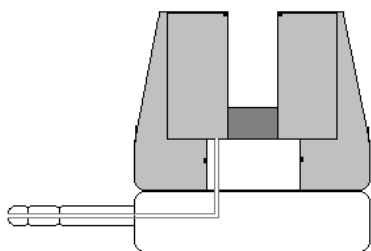
Instructions for use:



Overview of components

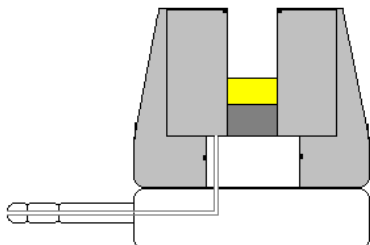
Step 1

Place one of the pellets in the cylinder with the optically polished surface facing up.



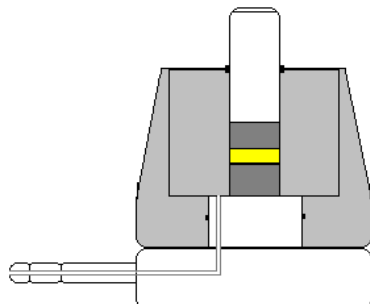
Step 2

Prepare and load sample into cylinder bore using a paper funnel or spatula. Use the plunger to spread and tamp the sample evenly over the pellet.



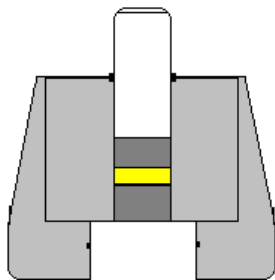
Step 3

Insert the second pellet with its optically polished surface facing down into the cylinder, followed by the plunger with O-ring. Place the die assembly onto the lower platen of your [laboratory press](#), connect a [vacuum pump](#) to the evacuation tube and evacuate the sample for two to three minutes. Apply load to the plunger (refer to the maximum load limit of the die) whilst continuing to evacuate the sample. Release the pressure when completed.



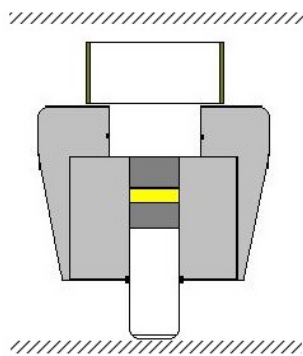
Step 4

Stop applying the vacuum and remove the evacuation tube with the die base.



Step 5

Turn the die upside-down and place it with the plunger resting on the lower platen of the laboratory press. Place the extraction ring between the upper platen of the press and the bottom of the die as shown in the illustration below. Apply a light load on the extraction ring until the pellets and sample disc are free of the cylinder.



Step 6

If necessary, place the sample disc into a [holder](#) – these are available for all standard sizes.



Step 7

Carefully clean all the parts of the die.

Dies with water jacket:

The water jacket allows heating (or cooling) of solid samples while under pressure when circumstances don't permit the use of electrical appliances.

Using a circulating water bath or pump, boiling water is introduced via the inlet tube into the jacket where it circulates before exiting via the other tube. (The third tube, shown at the bottom of the photo, is the vacuum connector in the base of the die, used for evacuating the sample). When using boiling water, the die body reaches ~80 to 100 °C, so wear thermal protection gloves if necessary.



Type of steel used for pellets:

The dies offered in our lab-club webstore are equipped with pellets made of SS-440C steel with a chromium content of 18%. They are hardened to about 60 Rockwell.

We also offer dies with tungsten carbide pellets containing 5% cobalt, making them approximately ten times harder than the SS-440C type. Please [contact](#) us for prices and delivery times.

Bear in mind that the hardness of the sample medium is not the primary consideration when deciding on the type of pellet to be used, rather it's the abrasiveness. Multiple use of a highly abrasive material will eventually scuff and damage the pellets and the inside wall of the cylinder.

KBr disc preparation for IR spectrophotometry:

A finely ground, approximately 1% mixture of a solid sample in KBr is fused into a transparent disc using a hydraulic press.

Any solid sample which can be reduced to a fine powder and which does not react with KBr can be examined using the pressed disc method. In addition to crystalline materials, rubbery or plastic samples can also be ground with KBr.

General Comments:

- Regular laboratory-grade KBr is likely to contain KNO_3 which gives spurious peaks. Don't use it.
- [Spectrophotometric-grade KBr](#) is completely transparent to infrared radiation, but is hygroscopic. Absorbed moisture causes broad water absorption bands near $3,333\text{ cm}^{-1}$ and $1,640\text{ cm}^{-1}$. Powder dampness is also evidenced by severe clumping of the powder.
- KBr should be oven dried overnight at approximately 110° C and then stored in a desiccator to eliminate moisture interference.
- Particle size of the sample affects the optical properties of KBr discs. Large crystals are very undesirable as they produce opaque spots. Extremely fine particles are also undesirable as they absorb water quite rapidly and tend to produce disc flaking.
- Uniform distribution of the sample in the KBr matrix is essential for achieving good results.
- A transparent disc is excellent. A translucent disc will work. An opaque disc will probably be useless.

Procedure:

1. Weigh exactly 1.5 mg (0.0015 g) of your dry, finely divided, solid sample into a small, clean [agate mortar](#). To ensure accuracy, weighing should be performed on an analytical balance with four decimal places.
2. Weigh 200 mg (0.20 g) of dried, spectrophotometric-grade KBr into the same mortar. If you use a polystyrene weighing boat, be sure not to scrape any of this into the sample as polystyrene is a strong IR absorber.
3. Using a clean pestle, grind and mix the mixture firmly and vigorously for several minutes or until the sample has been reduced to a very fine powder. At this point it should have a caked, glossy appearance. Work quickly and either wear a mask or avoid breathing on the sample whilst grinding – as mentioned above, KBr is hygroscopic.
4. The mixture is now ready for placing in your laboratory die.