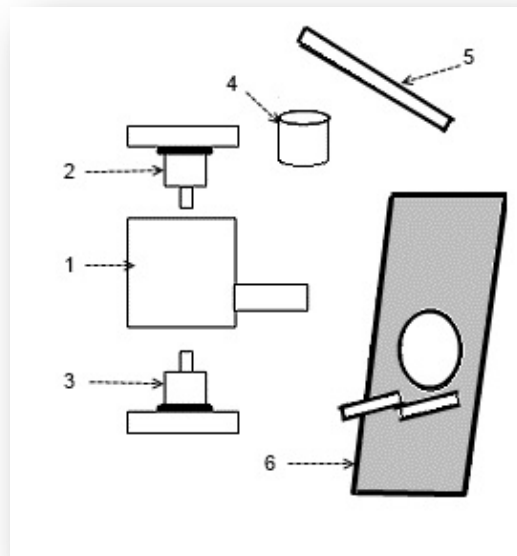


3mm evacuable die

- **Instructions for use**
- **Type of steel used for pellets**
- **KBr – general information and disc preparation for IR spectrophotometry**

List of parts:

1. Cylinder with vacuum connector
2. Stepped pellet
3. Stepped pellet
4. Filling funnel
5. Sample extractor
6. Die holder



Step 1

Place the cylinder with the vacuum connector at the bottom end on one of the pellets.

Step 2

Use the funnel to fill the cylinder with sample. Leave 1 to 1.5 mm free space for the second pellet.

Step 3

Place the second pellet on the top of the cylinder – the die is now complete. Attach the hose from your [vacuum pump](#) to the connector and place the die in the [laboratory press](#). Evacuate the die for 2 to 3 minutes. Continue applying the vacuum whilst applying the load to the die.

Step 4

When you have finished pressing, stop the vacuum pump and remove the hose from the connector.

Step 5

Take the die out of the press and remove both pellets. Place the cylinder on the two posts of the die holder and insert the holder into your spectrophotometer for analysis.

Step 6

Carefully clean all the parts of the die. If necessary, use the extractor to remove the sample.

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.