



Macro-Micro KBr Die

Operating Instructions

The Aldrich KBr Die, constructed of hardened stainless steel and aluminum, presses high clarity discs of materials such as potassium bromide, thallos bromide, and cesium iodide. The die produces 13mm discs, micro discs or pellets in paper inserts. The design and construction of the Aldrich KBr Die combines high performance, ease of maintenance and economy.

INSTRUCTIONS FOR ASSEMBLY, LOADING AND PRESSING

The proper assembly for the components of the KBR Die, during the pressing procedure, is illustrated in Fig. 1. *It should be noted that the anvil must be positioned with the polished side up.*

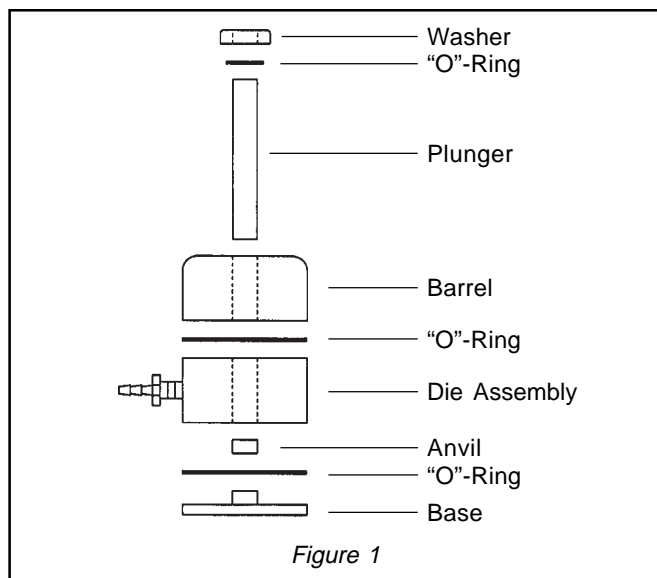


Figure 1

1. Premix the sample and matrix material, preferably in a mechanical vibrator. Place this mixture in cavity on the face of the anvil. *Mixing in the die should not be attempted.*
2. Level the sample and matrix material, using a microspatula or a clean, dry glass rod. *If surface of mixture is not leveled, opaque areas will be visible in the finished disc or pellet.*
3. Insert barrel into die, completing assembly as shown in Fig. 1. *Gently*, push plunger down to surface of powder for final leveling. Plunger should then be withdrawn approximately 1mm to 2mm.

CAUTION: Failure to observe the following steps could result in damage to the die. Read carefully!

4. Place the die in a press, assured that it is on a flat surface (not that of a soft metal), while pressure is being applied.
5. Connect vacuum line to die assembly. After two minutes under vacuum, apply pressure NOT IN EXCESS OF 12,000 POUNDS TOTAL LOAD (60,000 psi). Release pressure and vacuum after two minutes, and remove die from press.

Coherent discs will be produced starting at pressures of approximately 25,000 psi. In the case of potassium bromide, thallos bromide or cesium iodide, clear discs will generally not be produced until the pressure reaches 40,000 psi.

When using potassium bromide, approximate thickness can be determined from the fact that 300 to 400 mg will produce a 1mm disc, 13mm in diameter. The concentration required to produce good spectra in the 4000 to 600 wavenumber range is approximately one percent. Beyond 600 wavenumbers, a concentration of two to four times as much is required.

A useful conversion for a 13mm die: **pounds total load = .205 x psi**. For example, 25,000 psi is equal to 5,125 pounds total load and 40,000 psi is equal to 8,200 pounds total load.

INSERTS FOR MICRO PELLETS

Paper inserts with various apertures are available for use with the Aldrich KBr die to permit use of smaller amounts of matrix material and sample.

With round hole (diameter in mm)	With rectangular hole
1.5mm Z12,329-3	1.5mm x 9mm Z12,330-7
3.0mm Z12,332-3	
5.0mm Z12,333-1	
11.0mm ... Z12,331-5	

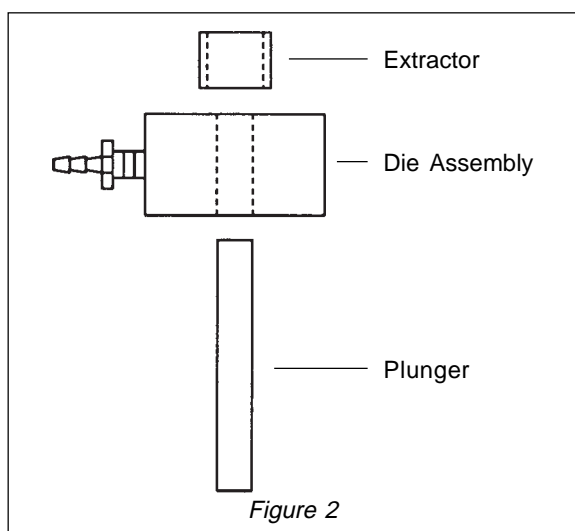
INSTRUCTIONS FOR PRODUCING MICRO PELLETS

Assembly of the die, for producing micro pellets, is the same as illustrated in the instructions for the 13mm disc, with the following exception: **The mixture will be carefully mounded, rather than smoothed prior to pressing.** The insert is placed on the polished surface of the anvil. The appropriate mixture of matrix material and sample is placed into the aperture and mounded carefully, as mentioned above.

The pellet is pressed under vacuum, using **one-fifth to one-fourth as much pressure.** The use of a 1.5mm pellet is recommended only in conjunction with a beam condenser.

INSTRUCTIONS FOR EJECTING A PELLET OR INSERT

1. After a pellet has been pressed, remove all components of the die except for those illustrated in Fig. 2.



2. Place extractor over aperture for ejection of pellet.
3. Put assembly into press.
4. Hold assembly with one hand while press is pumped and pressure, necessary, for ejection is applied.
5. As soon as it clears the well, anvil should be removed and the pumping rate slowed.
6. Transfer disc *immediately* to appropriate holder.
7. **Record spectrum as quickly as possible.**

SPECIAL PRECAUTIONS AND NOTATIONS

The Aldrich KBr Die is designed for pressing potassium bromide, thallos bromide and cesium iodide, but **cannot** be used for pressing silver chloride.

Do not use the die for pressing plastic pellets.

Thallos bromide is toxic and must be used with proper safeguards! These safeguards should be outlined in the literature which accompanies the material.

Somewhat lower pressures are required for producing CsI discs than for KBr and TlBr. These matrix materials all have different densities, refractive indices and transmission ranges. Only TlBr is not hygroscopic.

Pellets or discs pressed from cesium iodide must be pressed a second and third time. This is accomplished by leaving the pellet in the die for approximately five minutes, relieving the pressure of the press and then taking the pressure back up to 30,000 psi. The pellet is not removed until after the third pressing.

Making discs thicker than 1.25mm is not recommended.

INSTRUCTIONS FOR CLEANING AND STORAGE

KBr is a corrosive material which will rust stainless steel parts if not completely removed. Use a nylon bottle brush to scrub the well of the die. Wash the components with mild detergent and water. **Avoid washing compounds which contain tri-sodium phosphate.** Rinse die components with acetone and dry with a gentle stream of compressed air.

Store die components in a desiccator when not in use.