Evacuable Pellet Dies - User Instructions - 2I-03000-8

1. Introduction

The evacuable pellet dies available from Specac are manufactured in a wide range of diameters for both infrared and XRF applications. The standard dies, made from 440C stainless steel, offer an ideal solution for preparing sample discs/pellets prior to analysis. However, care must be taken where extremely hard, corrosive or irregular shaped samples are to be compressed, as stainless steel dies and pellets can be damaged.

All of the dies are evacuable, enabling dry, uncontaminated and clear pellets to be prepared.

2. Unpacking and Checklist

On receipt of your evacuable pellet die please ensure that the following have been supplied.

- 1 Die body (this has the evacuation port on dies of 20mm diameter and above).
- 1 Base (this has the evacuation support ring for dies up to 16mm diameter).
- 1 Plunger (this is stepped on dies smaller than 10mm diameter)
- 1 Pair of stainless steel pellets (unless stated)
- 1 Extractor ring (in Perspex, Delrin or aluminium depending on size of die).

Note: *Pellet/disc holders for mounting the sample disc in a spectrometer are not supplied with the dies but can be purchased separately from Specac. (e.g. P/N GS03410 for the 13mm diameter pellet.)*

3.Die Care and Use with KBr Powder

The dies are made from corrosion resistant steel, but because of the corrosive nature of wet KBr or possibly from other substances used with the dies, it is necessary to take certain precautions.

A) When not in use always ensure that the die and its component parts are thoroughly clean and dry. If possible, it is preferable to store the die and components in a drying cabinet or a desiccator.
B) When cleaning the die pellets, be careful of the polished faces. Do not use a cloth that has a hard abrasive texture. Always use a soft cloth or tissue. If KBr powder has been used as a sample for compression, any remaining powder should be washed away from the die body, plunger and pellets with distilled water and then rinsed with methanol. After drying the parts with a soft cloth they can be placed on a warming plate to keep warm and dry until use with the next sample.
C) Never exceed the maximum safe load when pressing.

Note: *KBr powder P/N GS03610 is supplied in a sealed glass bottle. The seal should not be broken until using the powder for the first time. Store the KBr powder in an oven at 50ºC or a desiccator to prevent absorption of moisture.*

4. Preparing the Evacuable Pellet Die for Use

The die and its component parts are packed at the factory in such a way that they do not suffer damage during transit. The optically polished pellets (**1**) are encapsulated in an easily removed protective material and the die may be lightly coated with a protective oil. Before use, the protective material on the pellets should be removed and all of the component parts must be thoroughly cleaned with an organic solvent (methanol is suitable) to ensure that the protective oil is fully removed. The parts should then be wiped clean taking care to use non-abrasive cleaning cloths on the polished faces of the pellets (**1**).

Place the base (**2**) of the die onto a work surface. (For dies up to 13mm in diameter the base will include a black coloured surrounding evacuation ring and port (**5**). For dies of 20mm diameter and above, the base does not have the surrounding evacuation ring and port. (The vacuum port is part of the die body). Ensure that the O-ring seal (**3**) is correctly positioned into its groove in the evacuation ring or around the smaller diameter piece of the base for dies of 20mm diameter and above.

5. Assembly for Evacuable Pellet Dies of 10mm Diameter and Above

Assemble the die cylinder body (**4**) onto its appropriate base (**2**) as shown in Figures 1 and 2. Figure 1 is for the 5mm, 10mm and 13mm dies with a black support ring for the base (**2**), carrying the evacuation port (**5**). Figure 2 is for the larger dies of 20mm and above, including the evacuation port (**5**) as part of the die body.

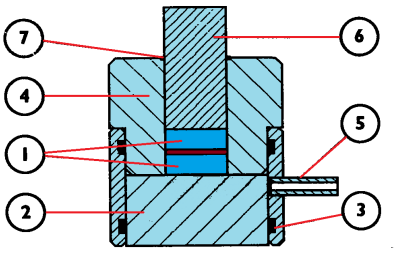

Figure 1.

Take one of the stainless steel polished pellets (**1**) and place it (polished side up) into the bore of the cylinder body (**4**). (Avoid getting fingerprints on the polished surface - wear gloves). The pellet is a very close fit in the bore but will eventually sink to the bottom of the bore in the cylinder body (**4**). (You can use the plunger (**6**) if required to aid in the travel of the pellet in the bore). When the first pellet (**1**) is in position, you can now fill the die with a prepared sample powder.

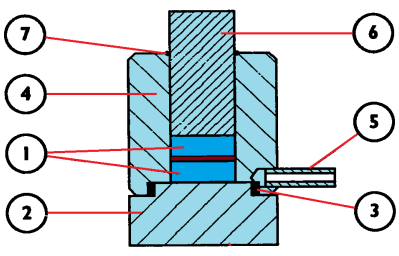

Figure 2.

6. Filling the Evacuable Pellet Die with Sample

This procedure is typically for the filling of a 13mm die, but can be adapted for your specific sample and size die. Using a paper chute, funnel or spatula, pour a well ground and mixed powder (2 to 4mg sample in 175 to 225mg KBr mixture) to be compacted into the bore of the cylinder body (**4**). Tap the side of the die lightly so that the powder is evenly distributed across the face of the polished pellet (**1**). You can also use a spatula to smooth over the surface if tapping of the die is insufficient.

Take the second stainless steel pellet (**1**) and push this (polished face first) into the bore of the cylinder body (**4**). Take care not to force or jam the pellet (**1**) in the bore. If it is placed centrally, it should sink down in the bore onto the surface of the powder. Insert the plunger (**6**), non- chamfered edge end first into the cylinder body (**4**). Ensure that the O-ring seal (**7**) is in place around the plunger and seated in the chamfer of the cylinder body (**4**) as shown in Figures 1 and 2. The die is now ready to be placed in the press for compaction of the sample powder.

7. Specific Assembly and Filling of the 5mm Evacuable Pellet Die P/N GS03060

Because of the stepped plunger (**5**) used in the dies smaller than 10mm diameter, as found in the 5mm die P/N GS03060, it is not easy to put the stainless steel pellets (**1**) into position with the die stood upright on its base. The whole operation of inserting the pellets (**1**) and filling with a sample is best done with the die inverted to allow access to the actual bore diameter. This may seem an awkward operation, having to hold the die steady in one hand whilst adding components from the other, but with practice it is achievable.

Before putting the cylinder body (**4**) on its base (**2**), take the plunger (**5**) and insert into the cylinder body (**4**) as normal. Holding the plunger into the die body, invert the assembly, and push a stainless steel pellet (**1**) (unpolished side first) into the bottom of the die cylinder body (**4**). Allow the pellet (**1**) to sink into the bore of the cylinder body (**4**) by pulling on the plunger, such that there is enough depth created in the bore to fill with a powdered sample. (See Filling the Evacuable Pellet Die with Sample). Fill the die with a sample. Now take the second stainless steel pellet (**1**) and push it (polished side first) into the cylinder body (**4**) bore such that the sample is contained between the two stainless steel pellets (**1**). Now, very carefully place the base (**2**) onto this inverted plunger/body/sample and pellets assembly such that the die can then be turned upright for pressing.

8. Pressing of the Sample Pellet

Place the die assembly into a hydraulic press. Make sure that the die is central on the bottom pressing face of the press.

If a vacuum supply is to be used on the die whilst pressing, connect the vacuum line tubing to the evacuation port (**5**) of the die. Switch on the vacuum line and maintain the vacuum before, during and after the pressing process.

Ensure that all safety guards on the press are closed and then start to press the die. Apply a load to the plunger (**6**) to produce the desired glassy quality of KBr/sample pellet. For the 13mm die, a load of 7 tons indicated at the pressure gauge of the press is usually sufficient.

Note: *The following table gives typical loads to produce good quality KBr pellets.*

Die Size	Typical Load	Maximum Load
5mm	1.25 tons	2.00 tons
10mm	3.00 tons	5.00 tons
13mm	7.00 tons	10.00 tons
20mm	18.00 tons	25.00 tons
32mm	25.00 tons	50.00 tons
40mm	25.00 tons	85.00 tons

Warning: DO NOT EXCEED THE MAXIMUM LOAD.
Hold the load on the die for 10 to 15 seconds and then release both the load and the vacuum.

9. Removing the Sample Pellet from the Die

This procedure is the same for all diameter size dies. Take the die out of the press and remove the base (**2**). All of the internal components in the die cylinder body (**4**) will remain in place due to compaction of the sample.

Invert the die and place the extractor ring (**8**) into position on the cylinder body as shown in Figures 3 and 4. (Figure 3 is for the 5mm, 10mm and 13mm dies and Figure 4 for the 20mm die and above).

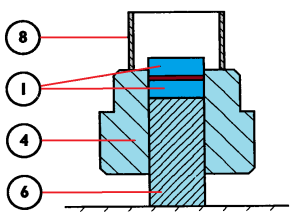

Figure 3.

Replace this inverted assembly into the press and apply a light load between the plunger (**6**) and the extractor ring (**8**). The load can be applied by rotation of the lead screw assembly in manual hydraulic or Atlas™ Series presses or by pumping the piston of the press. As the load is applied, the bottom stainless steel pellet (**1**) will emerge first from the bottom of the cylinder followed by the

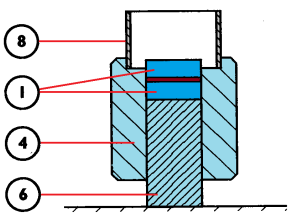

Figure 4.

compacted sample pellet and then the top stainless steel pellet. Usually, once the **sample pellet** has cleared the bore of the cylinder body (**4**), the plunger (**6**) will immediately pass through the bore to the limit of its travel, as the weight of the cylinder body sinks down due to no resistance.

Remove the die parts from the press and carefully extract the compacted sample pellet with a pair of forceps, taking care not to damage it in the process. The highly polished surfaces of the stainless steel pellets (**1**) aid in easy sample release from these pellets.

10. Paper Frame Mounting of Sample Pellets

To facilitate the handling of KBr sample pellets, paper frames can be used which are of a macro, micro or ultra-micro form.

The macro paper frame is a paper annulus of 13mm o.d. and 10mm i.d. These frames are to be used with the 13mm die only. When using these paper frames (9) they are inserted into the die prior to addition of the sample (**10**). When the sample pellet is extracted a transparent circular window in an opaque paper frame is produced.

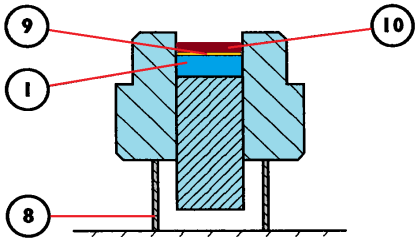

Figure 5.

The micro and ultra-micro frames are also circular with a 13mm o.d. but they have a rectangular aperture of 2mm x 11mm and 1mm and 4mm respectively. When using the micro or ultra-micro frames it is recommended that the die be loaded on the extractor ring (**8**) as shown in Figure 5. The die is assembled inverted with the pellet (**1**) and ultra micro frame in place. The aperture is carefully filled with the powder sample such that it is heaped 1 to 2mm above the surface of the frame (**9**). The bottom pellet (**1**) is then put into place and the base (**2**) is fixed to the cylinder body assembly. This whole assembly is then turned the right way up ready to be pressed.

11. Notes on Sample Pellet Quality

Generally, it is easy to produce a good quality pellet if the die is used correctly. However, some faults in the produced sample pellet may occur due to a variety of reasons. Some of these faults and their remedies are tabulated below. The faults described are for pure KBr or other halide salts, which do not contaminate the sample. When the sample is added to the halide salt the clarity of the disc will depend to a large extent on the quantity and type of sample. Usually 0.1 to 2% of sample to KBr is perfectly adequate. The overall quality of a pellet is largely dependent upon the quality of the KBr or halide salt powder used, which should always be of a spectroscopic grade of purity.

Fault	Cause	Remedy
Sample pellet not clear. Lacks optical clarity.	Sample damp, contaminated KBr powder or insufficient pressure when compacting.	Dry the KBr powder or sample and increase the compacting pressure.
Sample pellet is clear but shows opaque spots.	Powder not uniformly flat in the die, leaving large particles which do not vitrify when pressed.	Sieve powder to extract coarse grains, then re-grind and re-press.
Sample pellet is cloudy.	Insufficient evacuation time or leaky seals.	Check seals on the die and lengthen evacuation period.
Sample pellet is clear at first but quickly becomes cloudy.	Damp powder or damp atmosphere.	Dry the KBr powder or sample, check seals on the die and lengthen evacuation period.

To ensure that a sample pellet is produced which will enable accurate spectra of samples to be obtained, it is essential that the sample be thoroughly blended with the halide salt powder. Blending can be achieved either by using a mortar and pestle (P/N GS03600) or by using a grinding mill such as the Specamill (P/N GS06000).

12. Legend

(**1**) Polished pellet
(**2**) Die base
(**3**) O-ring seal in evacuation ring
(**4**) Die cylinder body
(**5**) Evacuation port on die
(**6**) Plunger
(**7**) O-ring seal for plunger
(**8**) Extractor ring
(**9**) Paper support frames
(**10**) Sample

13. Spare Parts for Evacuable Pellet Dies

All of the die parts are available for individual replacement. Because of the wide range of die sizes and parts please contact Specac for any replacement parts you may require for your specific die size.

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Eurolabo - Paris.
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