

Solid and ATR Kit – Shimadzu - China

User Manual

2I-01139-4

Solid and ATR Kit – Shimadzu – China

P/N GS01139

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1. Introduction

Thank you for buying a product from Specac.

The Solid and ATR Kit – Shimadzu - China P/N GS01139 consists of the Specac Mini-Pellet Press P/N GS03940 provided with a 7mm Pellet die assembly P/N GS03950 and a 3" x 2" slide mount holder P/N GS03960, for solid sample preparation and their analysis by formation of a 7mm diameter KBr pellet.

Included with the Mini-Pellet Press components under P/N GS01139 is the Quest ATR Accessory offered as P/N GS10802-B. This consists of the Quest ATR optical unit with black coloured surface and clamp anvil tower assembly with a ZnSe crystal puck assembly and a Shimadzu Benchmark baseplate (P/N GS49103).

The Specac Mini-Pellet Press and the Quest ATR Accessory are provided in their own separate cardboard carry cases. Both forms of packaging include a respective instruction manual for use and explanation of the accessory within.

This instruction manual for P/N GS01139 explains how the Mini-Pellet Press and its 7mm die assembly is used and how a 7mm diameter KBr pellet is prepared for subsequent analysis by the infrared transmission spectroscopic technique

The instruction manual supplied with the Quest ATR Accessory for P/N GS10802-B explains how this accessory is used and how different solid and liquid samples can be handled for analysis by the infrared ATR spectroscopic technique. A separate instruction for installation of the Shimadzu Benchmark baseplate to mount the Quest ATR Accessory is also provided with the Quest ATR Accessory.

Mini-Pellet Press Introduction

The Specac Mini-Pellet Press, together with its own 7mm Pellet Die assembly allows for the production of 7mm diameter KBr pellets for analysis of solid samples by FTIR spectroscopy. These items are an

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alternative to the use of, typically, a 13mm diameter pellet die assembly within a 15 ton manual hydraulic press to produce 13mm diameter KBr pellets/discs.

The Mini-Pellet Press has been designed to produce a maximum 2 tons load which is sufficient to provide a force for formation of excellent quality KBr pellets with a 7mm diameter. When the 7mm pellet die assembly with a KBr sample has been placed in the Mini-Pellet Press, a 2 tons load can be easily and quickly applied by turning of a pressure screw hand knob. This action compresses a hydraulic fluid (oil) to raise a pressing piston and compress a sample held in the pressing area. To assist in the turning of the pressure screw knob an M8 (6mm A/F) size short handled Allen key is included to fit the central socket screw for greater leverage if required. To help in knowledge of the load being applied there is a small pressure indicator gauge on the press body which shows the actual pressure being applied and when to stop turning the hand knob.

The Mini-Pellet Press is small and relatively light enough in weight that it could be held in the hand to turn the pressure screw hand knob if desired, but for more secure use the press has been designed to sit and work on a bench surface.

Warning: *If you use the Mini-Pellet Press near the edge of a bench surface to turn the pressure screw knob, be careful that as much of the weight of the press as possible is on the bench surface itself, to prevent the press from falling to the floor.*

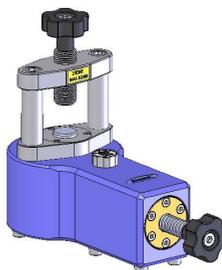


There is no requirement to permanently bolt the press to a work surface to apply a load safely to a sample, thus enabling easy storage of the press if needed.

2. Checklist of Contents

Check that the following items have been supplied with the Solid and ATR Kit – Shimadzu - China under P/N GS01139 for the Mini-Pellet Press components.

- Mini-Pellet Press
- P/N GS03940.



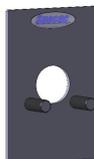
- 7mm Pellet Die assembly
(including its own Pellet Ring Holder) – P/N GS03950.



- Two spare Pellet Ring Holders
– P/N GS03951.



- 3" x 2" slide mount plate to
hold 7mm Pellet Ring Holder
– P/N GS03960.



- Pestle and Mortar P/N GS03600
- Bottle of KBr powder (50g) P/N GS03610
- M8 (6mm A/F) short handled Allen key

Carefully remove the Mini-Pellet Press, die parts and other items supplied with the Solid and ATR Kit – Shimadzu - China and prepare the items for use.

3. Operation of the Mini-Pellet Press

Making a 7mm KBr pellet is simple and quick. The 7mm pellet die assembly (1) is easily located within the pressing area of the Mini-Pellet Press (2). The lead screw assembly (3) is turned until hand tight to secure the 7mm pellet die assembly (1) in position in readiness to turn the pressure screw hand knob assembly (4). **Only a light force is needed** to turn the pressure hand knob (4) to provide a sufficient tonnage load, but the M8 Allen key provided can be used to turn the central socket screw (5) for greater leverage if desired.



Note: NEVER use any other tool than the supplied M8 Allen key to turn the pressure hand knob (4) if hand tightening alone is insufficient to provide a maximum of 2 tons load.

The produced 7mm KBr pellet is retained within the pellet die ring holder body (6) and this is transferred to a 3" x 2" slide mount (7) to fit in the spectrometer. (See Fig 1.)

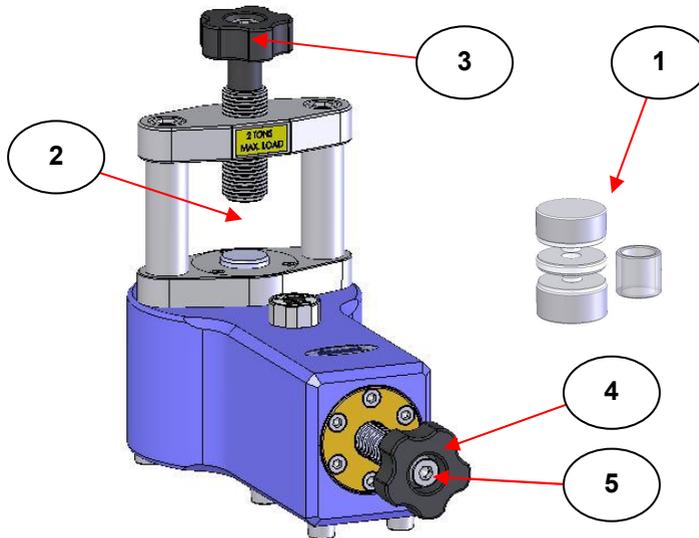


Fig 1. Mini-Pellet Press and 7mm Pellet Die Assembly

Preparation of the 7mm Pellet Die Assembly

Prior to placing the 7mm pellet die assembly (1) into the Mini-Pellet Press pressing area (2), a potassium bromide (KBr) powder mixture with a solid sample is prepared. A suitable quantity of the solid sample with an amount of KBr powder (P/N GS03610) in the usual ratio of 1 part sample to 300 parts KBr can be ground together using the agate pestle and mortar supplied (P/N GS03600) or an automatic grinding mill such as the Specamill (P/N GS06000) with an agate capsule set (P/N GS06200).

The 7mm pellet die assembly complete (1) consists of a central pellet ring holder (6), top anvil (8), a bottom anvil (9) and a clear Perspex extractor ring cap (10). (See Fig 2.) The top anvil (8) has a longer central “plunger” section than the bottom anvil (9).

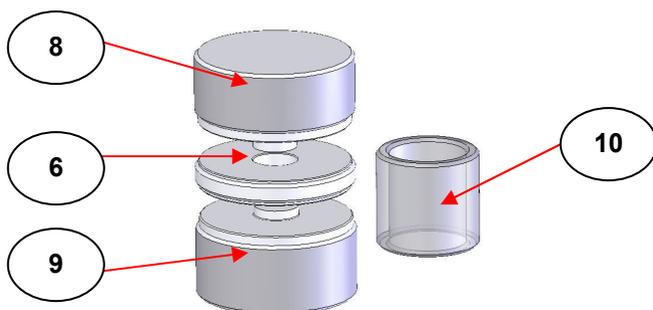


Fig 2. 7mm Pellet Die Assembly Complete

When the sample has been ground sufficiently, place some of the KBr mixture into the central hole of the 7mm pellet die ring holder (6) as it rests on the bottom anvil (9). (see Fig 3.)

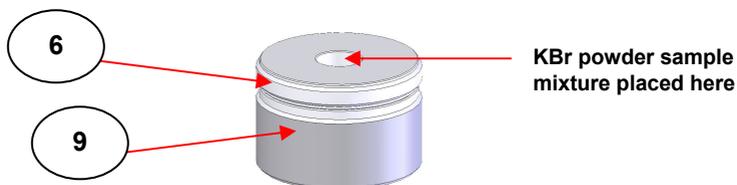


Fig 3. 7mm Pellet Die Ring Holder (Body) on Bottom Anvil

Fill the aperture hole of the 7mm pellet die ring holder (6) with sufficient KBr powder mixture to reach the top. Using a flat blade or the edge of the spatula, level off the powder mixture such the top of the powder mixture in the hole is flush with the 7mm pellet die ring holder's (6) top surface.

Now, take the top anvil (8) and place it carefully (plunger face downwards) over the powder/7mm pellet die ring holder (6)/bottom anvil (9) assembly. Locate the central plunger section of the top anvil (8) over the powder sample within the aperture hole and use light pressure from your hand to compact the powder mixture into the hole. The mixture is sufficiently soft to allow the plunger part of the top anvil (8) to fit and locate loosely into place in the aperture hole of the 7mm pellet die ring holder (6).. (See Fig 4.)

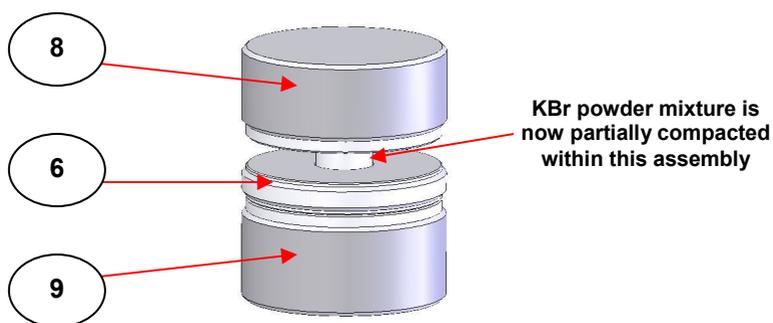


Fig 4. 7mm Pellet Die Assembly with Sample Prior to Pressing

When this stage has been reached you can carefully transfer this complete 7mm pellet die assembly (with sample) into the Mini-Pellet Press for full pressing of the KBr powder sample.

Note: *The bottom anvil (9) has a recessed underside surface with a metal rim around its circumference. The recess allows for central and correct positioning of the 7mm pellet die assembly (1) over the piston (11) of the Mini-Pellet Press prior to any pressing.*

Pressing the 7mm Pellet Die in the Mini-Pellet Press

Prepare the Mini-Pellet Press to accept the 7mm pellet assembly (1) as formed at the stage shown to Fig 4.

The lead screw (3) on the Mini-Pellet Press is retracted (turned anticlockwise) so there is sufficient “daylight” between the pressing face of the leadscrew (12) and the piston (11) of the Mini-Pellet Press. The piston (11) will be at its rest position for the start of its travel. This will be when there is no applied pressure on the system from the pressure hand knob (4) or shown at the pressure/load gauge (13). (See Fig 5.)

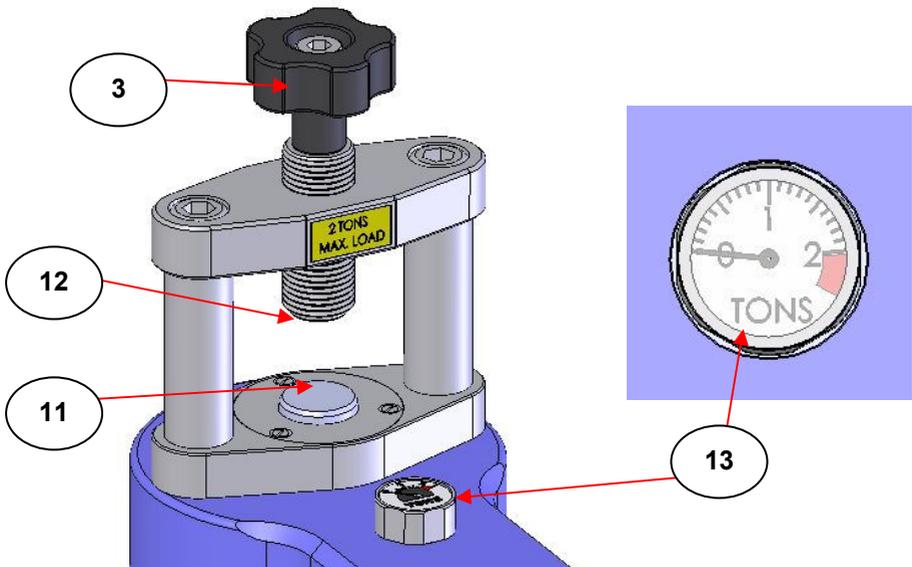


Fig 5. Mini-Pellet Press Piston Area and Pressure Gauge Reading Before Applying a Load

Before applying any pressure (load), to the die assembly (1) when it is placed into the press, the pressure screw hand knob assembly (4)

should be fully unwound to its start position. Turn the knob (4) anticlockwise until it will turn no further such that the screw assembly has reached its end stop. When the pressure screw hand knob assembly (4) is fully retracted, the pressing piston (11) will be at its correct start position for pressing.

When the Mini-Pellet Press pressing area has been set to accept the 7mm pellet die assembly (1), place the die assembly carefully and centrally into the press and loosely clamp the die assembly into position by turning the lead screw assembly (3) clockwise. (See Fig 6.)

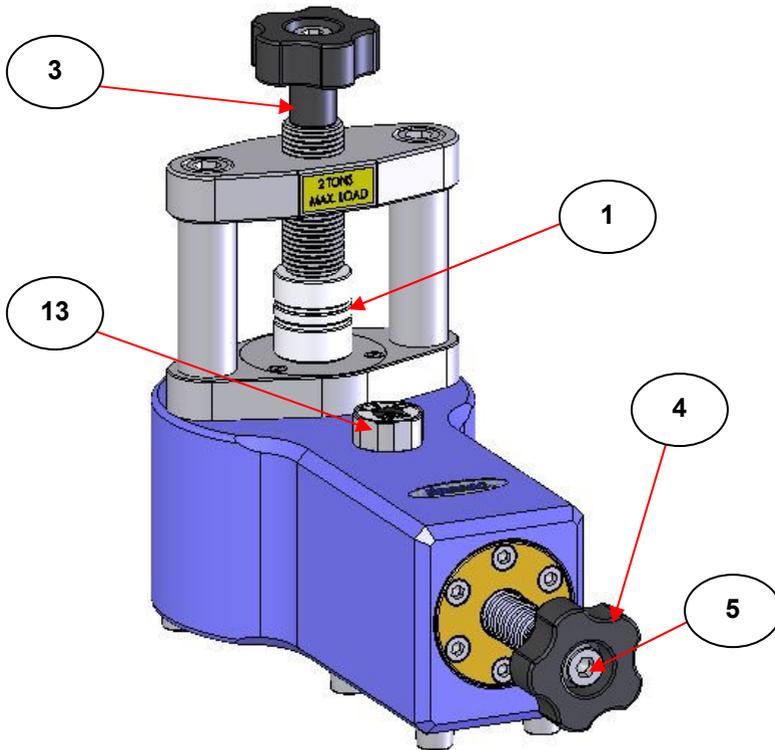


Fig 6. Mini-Pellet Press with 7mm Die Assembly in Position

Before turning the pressure screw knob assembly (4) to apply a load, tighten the lead screw handle assembly (3) a little further clockwise to continue the initial compaction of the KBr powder mixture contained within the die. When the lead screw (3) can no longer be hand tightened, the KBr powder mixture is ready to be fully compressed by application of a load provided by turning of the pressure knob screw assembly (4). Begin applying a load by turning the pressure knob (4) clockwise. It will be easy to turn at the start, but gets progressively harder to turn as the pressure (a tonnage load) increases and is being applied to the die assembly (1). (Use the M8 Allen Key supplied if required in screw (5).) The increase in pressure will be indicated by the pressure gauge (13) on the Mini-Pellet Press body.

Continue turning the pressure knob (4) until the pressure gauge (13) needle is pointing at the 1.75 tons division indicator. At this tonnage reading stop turning the pressure screw knob assembly (4). This will be a sufficient tonnage load to have compacted the KBr powder together to form a suitable pellet.

Note: *On the pressure gauge (13) there is a red colour warning area beyond the maximum 2 ton load reading. **NEVER** over-pressurise the Mini-Pellet Press such that the indicator needle has entered this red colour section of the pressure gauge.*



Removing the 7mm Pellet Die Assembly from the Mini-Pellet Press

When the 7mm pellet die assembly (1) has been compressed to a sufficient tonnage load to form a solid KBr pellet within the pellet die ring holder (6), the die assembly (1) can be removed from the Mini-Pellet Press.

Remove the pressure (tonnage load) from the pellet die assembly (1) by anticlockwise rotation of the pressure screw knob assembly (4). When the pressure gauge (13) indicator needle drops to zero tons the 7mm pellet die assembly (1) should be free to be removed from the pressing area (2). However, it is always best to fully retract the pressure screw knob assembly (4) to its end stop, to ensure that the piston (11) returns to its correct start/rest position for a new pressing.

Positioning the KBr Pellet Die Ring Holder in the Spectrometer

Having removed the compressed 7mm pellet die assembly (1) from the Mini-Pellet Press, the top anvil (8) and bottom anvil (9) are separated/removed from the die ring holder (6). Hold the knurled outer edge of the die ring holder (6) with one hand and twist/turn the anvils (8 and 9) by holding their knurled edges with the other hand. The KBr/sample powder material will have been compacted into a 7mm diameter pellet contained in the die ring holder (6) central aperture hole.

The complete die ring holder (6) with KBr pellet is now placed into an infra red spectrometer system by use of the 3" x 2" slide mount holder (P/N GS03960) (7). The die ring holder (6) rests simply on the two support rods of the 3" x 2" slide mount holder (7) and is held tight against the 3" x 2" slide plate by compression of the two O-rings (14) supplied having been slid over the support rods as shown. (See Fig 7.)

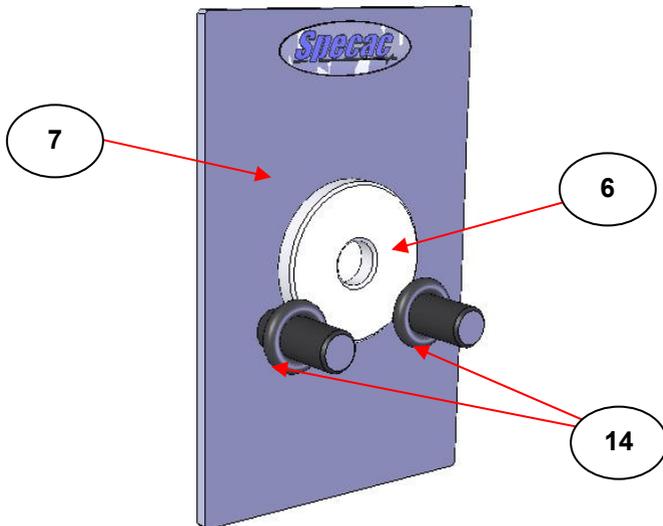


Fig 7. 7mm Pellet Die Ring Holder on 3" x 2" Slide Mount

Cleaning of Pellet Die Parts and KBr Pellet Removal Intact from the Pellet Ring Holder

When the KBr pellet sample being held in the die ring holder (6) has been analysed, the KBr sample must be removed from the die ring holder (6) for pressing of a new sample into the same die ring holder.

Note: *There are two spare die ring holders (6) P/N GS03951 provided with the Basic Solid Pack to use for preparation of a second or third sample, if the die ring holder (6) of the pellet die assembly (1) is already being used for spectral data collection.*

To remove the KBr pellet, the entire KBr pellet and die ring holder (6) assembly can be rinsed with cold or warm water to gradually dissolve the KBr sample. When the KBr sample has been washed away from inside the holder, the die ring holder (6) part can be further washed/rinsed with methanol and then dried with tissues. Specac would also then recommend that this part is stored in dry environment or placed on a heated surface (circa 30°C to 35°C) prior to use for the next time. After initial pressing of the pellet into the die ring holder (6) (from sample preparation stage), the top anvil (8) and bottom anvil (9) should also be rinsed with water, then methanol, dried with tissues and stored on a heated top plate surface ready for next use.

Note: *Thoroughly clean any KBr residues away from the die and press parts (screw threading's) to keep these items in working order.*

However, if you would wish to **save** the 7mm KBr pellet that has been formed within the die ring holder (6) after its spectroscopic analysis, then this KBr pellet can be removed intact from the die ring holder (6) part, by use of the top anvil (8) with the longer plunger and the Perspex die extractor cap piece (10).

Take the die ring holder (6) with KBr pellet and place the plunger of the top anvil (8) into the aperture hole on one side of the die ring holder (6). Place this combined assembly with the Perspex extractor cap (10) in contact with other side of the die ring holder (6) into the Mini-Pellet Press pressing area (2) as shown in Fig 8. Make sure that this assembly of parts is placed centrally between the lead screw pressing face (12) and the piston surface (11).

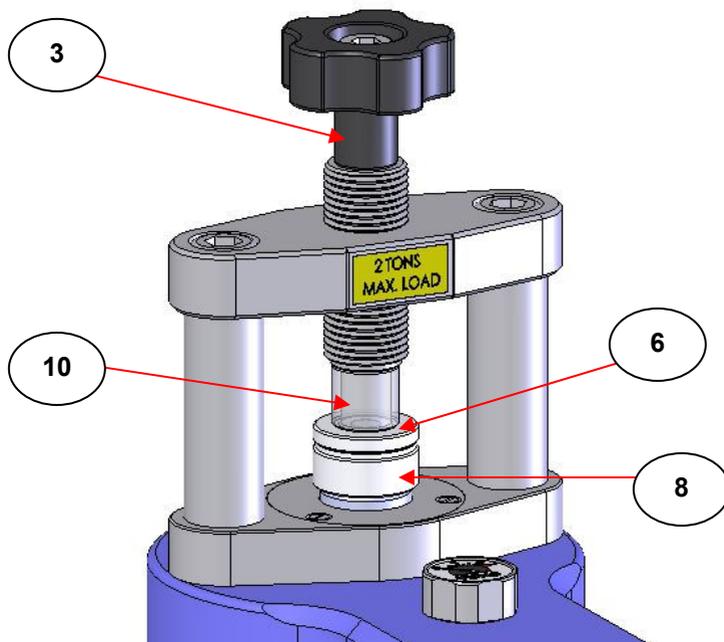


Fig 8. 7mm Pellet Die Ring Holder, Top Anvil and Extractor Cap Assembly in Position for KBr Pellet Extraction

Loosely hold the components together by turning the lead screw assembly (3) clockwise and then continue turning the lead screw assembly (3) to allow the top anvil (8) to push the KBr pellet out of the die ring holder (6) and into the space created by the Perspex extractor cap (10). When the KBr pellet has been pushed out of the die ring holder (6), retract the lead screw assembly (3) by a couple of anticlockwise turns and carefully remove the assembly of parts from the Mini-Pellet Press pressing area (2) to gain access to the KBr pellet.

The intact KBr pellet can now be saved or used for any further purpose and the die ring holder (6) and top anvil (8) parts can be cleaned (water and then methanol rinses, then drying and placed on a hot surface) as described previously, ready to be used again for the next KBr sample pressing.

4. Spare Parts for the Basic Solid Pack

P/N GS03940 – Mini-Pellet Press.

P/N GS03950 - 7mm Pellet die assembly complete.

P/N GS03951 - 7mm Pellet die ring holder (body).

P/N GS03952 - 7mm Pellet die top anvil (long plunger).

P/N GS03953 - 7mm Pellet die bottom anvil (short plunger).

P/N GS03954 - 7mm Pellet die Perspex extractor cap.

P/N GS03600 - Pestle and Mortar.

P/N GS03610 - KBr powder (50g).

P/N GS03960 - 3" x 2" slide mount for 7mm pellet die ring holder.

5. Legend for the Basic Solid Pack

- (1) 7mm Pellet die assembly.
- (2) Pressing area of Mini-Pellet Press.
- (3) Lead screw assembly.
- (4) Pressure screw knob assembly.
- (5) Pressure screw knob assembly.
- (6) Pellet die ring holder (body).
- (7) 3" x 2" slide mount.
- (8) Top anvil (long plunger).
- (9) Bottom anvil (short plunger).
- (10) Perspex extractor cap.
- (11) Lead screw pressing face.
- (12) Piston.
- (13) Pressure gauge.
- (14) O-rings for ring holder fixing when fitted on 3" x 2" slide mount.

Specifications of the Mini-Pellet Press

Maximum load - 2 tons.

Pellet Die diameter 7mm.

Maximum space between pressing faces - 45mm.

Minimum space between pressing faces - 15mm.

Piston stroke - 0.5mm.

Upper pressing face area (lead screw diameter) - 17.0mm.

Lower pressing face area (piston diameter) - 21.6mm.

Dimensions (excluding leadscrew assemblies) - 110mm wide x 200mm long x 155mm high.

Dimensions (including leadscrew assemblies) - 110mm wide x 265mm long x 215mm high.

Weight - 4.2Kg.

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