

Best practice and safe operational procedure for pellet dies

Before using the dies prospective users in NAFUMA must be trained by equipment responsible Rasmus Vester Thøgersen (ØU-37, rasmusvt@smn.uio.no, 901 82 759).

Members of other groups must contact Anja O. Sjøstad (Ø-130, a.o.sjastad@kjemi.uio.no, 905 01 603) for authorisation and to schedule training and use.

1) Use safety glasses

2) Make sure you choose a die suited for your purpose. Some users are more dependent on a clean and high quality pellet, and contamination can be detrimental to their work. Please do not use our best dies unless absolutely necessary. If unsure, ask!

3) Inspect that the die is clean and that all parts are in good conditions prior to any use. If cleaning is required, see step 8. Please notify equipment responsible if conditions are unacceptable.

4) Start with dry and well ground powders – the smaller and more homogenous the particle size of the powder the better. The increased surface area of the small particle enhances diffusion between particles during sintering. Hand grind thoroughly with a mortar and pestle, or use ball-milling to achieve this. The smaller particles will also pack together more closely, so the initial pressed pellet will be more dense and robust.

5) Assemble die and loading of powder (Figure 1)

- Use a Q-tip to smear the inner wall of the body (B) with 2.5 wt% stearin in isopropanol. Let dry before filling the powder. You can find the stearin in a bottle on the shelf. If the bottle is getting close to being empty, contact the equipment responsible so that it can be refilled.
- Assemble body (A) and base (B)
- Insert one metal pellet (E) with the shiny side pointing upwards in the base hole. Avoid fingerprints on the shiny surface – use gloves!
- Load the well ground powder into the bore of the die (inside B), but leave a space corresponding to the height of two metal pellets or more. Make sure there is no powder on the side of the bore. If there is, gently clean this with a Q-tip while being careful not to touch the sample in the bottom of the bore.
- Insert the second metal pellet (E) with the shiny side pointing downwards in the hole. Don't force it in, it should go down by itself, or you could use the weight of the plunger (see next point) to get it in.
- Insert the plunger (D) with the flat side going into the body, and the truncated side pointing upwards.

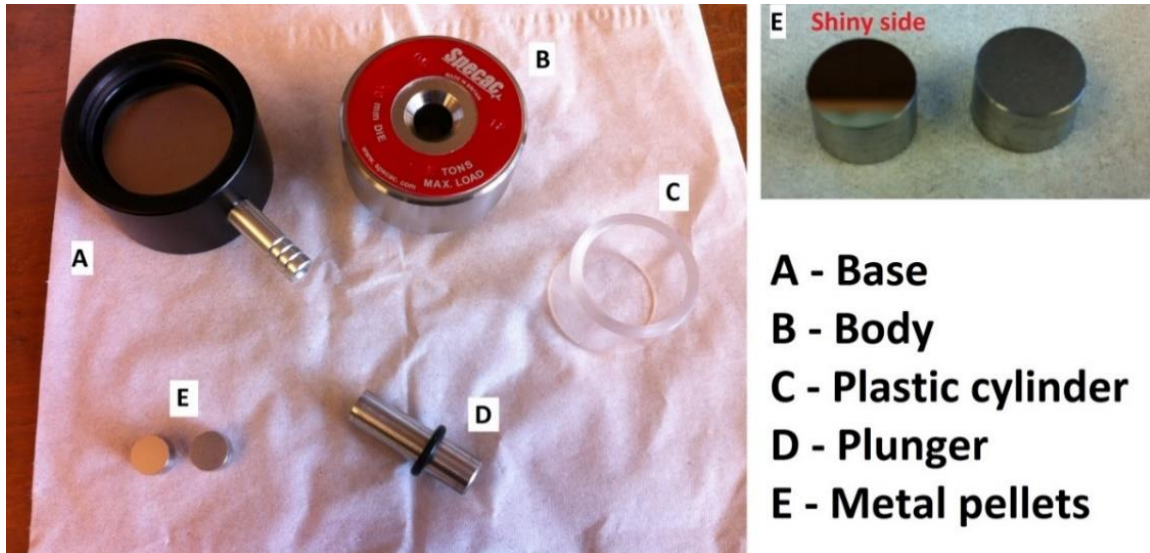


Figure 1: Picture of the die parts.

6) Press powder into pellet

- Make sure that valve 1 on the pump is closed. You close it by turning it clockwise (away from you). (Figure 2)
- Build up the press platform using metal cylinders so that the distance between the top of the die and the metallic press frame is as small as possible (see Figure 3)
- Hold the die carefully in position in the press while slowly pumping the pressure up to **maximum** allowed operating pressure of the hydraulic press (200 bar/3000 psi for metal dies with diameter larger or equal to 10 mm; 70 bar/1000 psi for the 5 mm die). **Be aware, using the highest allowed load of the hydraulic press does NOT always result in a better pellet, and typical loads are 60-70% of maximum.**
- Release pressure on the hydraulic press by opening valve 1 on the pump. You release by turning it anti-clockwise (towards you). (Figure 2)



Figure 2: Picture of the pump.



Figure 3: The die mounted on the press platform, and on the table are the rest of the metal cylinders available.

7) Removal of pressed pellet from the die

- Remove the die from the press platform
- Dismount body from base. Be aware that the bottom metal pellet may be loose
- **If possible, eject the pellet manually. If not possible, proceed with the steps below.**
- Mount the body (including the plunger, pressed sample and metal pellet(s)) back in the hydraulic press with the plunger pointing toward the press platform. Place hollow plastic cylinder (C) on top of the body – assure correct alignment of the plastic cylinder (Figure 4)
- Hold the body carefully in position in the press while pumping slowly until the metal and sample pellet are released from the body. In case the height of the metal and sample pellet is larger than the height of the plastic cylinder stop pumping when the metal pellet is released. Remove the metal pellet and repeat the procedure to remove the sample pellet.

8) Cleaning of die

- **Clean the die between each individual pellet, even if pressing the same material!**
- In the cleaning procedure it is:
 - **strictly forbidden to use any sharp tools or objects to scrape off powder inside the body**
 - **only allowed to rinse with water and isopropanol/ethanol. Avoid all acidic and basic liquids as these may easily deteriorate the die**
 - **only allowed to use the cloth/cleaning materials cotton, Q-tips and Kleenex**
- Dismount all parts of the die
- Rinse all parts carefully with hot water
- Dry and polish all parts carefully with cotton/Kleenex paper/Q-tips
- Finish the rinsing process by washing and polishing all parts using ethanol or isopropanol, and using the hair drier to remove all liquid before packing the die away or reusing it to make a new pellet.
- Carefully inspect that all powder residues are fully removed.
- In case you are unable to remove all residues of your sample from the die, **contact equipment responsible immediately! Do not leave the equipment dirty!**



Figure 4: Pictures of how the removal of the pressed pellet should be performed.

9) Clean up the work station and leave it in the same conditions as it was when you arrived.

- The metal pellets should be wrapped in a Kleenex tissue, but NOT in contact with each other, and kept in the plastic cylinder.
- The body and base should be stored assembled, with the metal plunger inside.
- The die should be kept in its designated cardboard box together with the metal pellets, and be placed in the appropriate plastic container.