Standard Operating Procedure

Cold Seal

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| **Department:** | Earth and Environmental Sciences |
| **Date SOP was written:** | 9/19/2013 |
| **Date SOP was approved by PI/lab supervisor:** | 9/19/2013 |
| **Principal Investigator:** | John Ayers |
| **Internal Lab Safety Coordinator/Lab Manager:** | Richard Bradshaw |
| **Lab Phone:** | Click here to enter text. |
| **Office Phone:** | J. Ayers (615) 322-2158 (campus phone: 2-2158) Bradshaw (615) 343-0839 (campus phone: 3-0839) |
| **Emergency Contact:** | J. Ayers (615) 973-1879  R. Bradshaw (208) 260-2792 |
|  |
| **Location(s) covered by this SOP:** | *SC 5712* |
| *(Building/Room Number)* |

**Type of SOP:** ☒ Process ☐Hazardous Chemical ☐ Hazardous Class

**Purpose**

**Personal Protective Equipment (PPE)**

**Respirator Protection**

**Eye Protection**

ANSI approved, tight-fitting safety glasses/goggles and/or face shield.

**Body Protection**

Remove any loose jewelry around your neck or any jewelry on your hands and wrists. Tightly secure long hair in ponytail.

**Ear Protection**

Ear protection in the form of ear muffs or ear plugs must be worn during operation.

**Engineering Controls**

**Medical Emergency**

To contact the [Vanderbilt University Police Department](http://police.vanderbilt.edu/) in an emergency:

* Call **911** from any campus phone.
* Call **(615) 421-1911** from any other phone.

**Protocol/Procedure**

**Report any problems or abnormalities with the equipment immediately to the PI or LM.**

**\*\*\*NEVER leave the machine unattended while operating\*\*\***

**Loading samples**

1. Clean the inside of the vessel with deionized water and a brush
2. Lubricate the screw thread (a) of the vessel with grease
3. Insert the capsule (b) in the open space at the end of the filler rod (c), with the top of the capsule as in Fig. 1.
4. If the capsule is shorter than the open space in the filler rod, add a piece of scrap noble metal tube (d), in order to keep the capsule in the hot spot of the furnace. Cut scrap tube and then fold it around the thin wall of the filler rod as in Fig. 1. Other material can be used as long as it is strongly attached to the filler rod in order to avoid it getting stuck when the filler rod is retrieved at the end of the run.
5. Place the filler rod with the capsule into the vessel, introducing the end of the filler rod with the capsule first. Push the filler rod to the bottom of the vessel.
6. Usually, the filler rod is shorter than the length of inner space in the vessel. Consequently it is necessary to fill the resulting space in order to keep the capsule in the hot spot. The supplement material (e) should allow the free circulation of pressure fluid into the vessel. A piece of scrap noble metal tubing can be used. Note: since the closure-nut assembly (f) is a cone-in-cone system, be sure do not block the cone shape space of the vessel with the supplement material (e).
7. Fill the space between the filler rod and the wall of the inner part of the vessel with deionized water. Shake it a little to allow air bubbles to come out.
8. Check in the closure-nut assembly (f) that the inner adjustable nut (g) allows the cone-end of the pipe to protrude like in the Fig. 1. Avoid a situation as in (h).
9. Keep the vessel vertical and carefully place the closure-nut assembly on the screw thread (a) of the vessel and tighten it by hand, then tighten by using two wrenches like in (i). Alternatively, if you find it difficult to deal with two wrenches at the same time, you can put the vessel in a clamp and tighten the closure-nut with a wrench. Remember to keep the vessel in a vertical position.
10. Check the bottom-nut assembly (l) so that the inner adjustable nut (m) allows the cone-end of the pipe to protrude as in the Fig. 1. Avoid a situation like in (h).
11. Add deionized water to the space in (k) Fig. 2 before placing the vessel and then tighten it as in (j) Fig.1, Note: try to maintain the external-thermocouple well (r) of the vessel in a forward position, as in Fig. 2, in order to facilitate subsequent external-thermocouple (s) insertion.
12. Close the pressure valve (key) (o) Fig. 2
13. Turn on the air-compressor. When 80 psi is reached (on the 0 to 160 psi manometer) of the water pressurizing apparatus.
14. Then open the pressure valve (key) to pressurize the main line up to 5000 psi (on the 0 to 30000 psi manometer). Check the main line for leaks.
15. With 5000 psi in the main line, open the pressure valve (o) Fig.2 allowing pressurization of the vessel. Pressure in the vessel does not build up until all the air in the pressure inlet pipe (p) has been dissolved into the liquid.
16. Consequently, close the pressure valve (key) (o) and repeat steps 14 and 15 until you get 2000 or 3000 psi (manometer (q) Fig. 2), then close the pressure valve (key) (o).
17. The external-thermocouple (s) must be securely installed in order to avoid problems when furnace (t) is dropped. In addition, check the length of the naked end (u) of the external-thermocouple, it must be long enough to reach the bottom of the vessel well (r). Tie tightly the sheathed wires (v) of the external-thermocouple to the closure-nut (f) as in (w) Fig. 1.
18. CAREFULLY drop the furnace, to avoid crashing the ceramic rod insulator or moving the external-thermocouple position. The full length of the vessel (with the exception the closure-nut (f)) must be inside the furnace, in order to keep the capsule (b) in the furnace hot spot. If it is not possible to introduce the vessel completely in the furnace, check the position of the furnace thermocouple (x) inside the furnace.
19. Set the temperature of the experiment on the temperature controller (y) Fig 2, and then turn on the power key (z).
20. Pressure in the vessel is going to start building up as the temperature rises. To avoid exceeding the maximum pressure limit of the vessel or the pressure of the experiment, monitor the pressure on the manometer (q) until the set temperature (step 19) is reached.
21. If the pressure achieved is higher than the pressure desired for the experiment, release it by opening the pressure valve (key) (o) very slowly. Note: any sudden change in pressure at high temperature can potentially damage the capsule (b).
22. In practice, it is much easier to achieve the desired pressure by progressively releasing excess pressure (as in step 21), rather than trying to increase a low pressure, by pumping fluid to the vessel, specially in the range over the 10000 psi.

**NOTE**

Any deviation from this SOP requires approval from PI.

**Documentation of Training** (signature of all users is required)

* Prior to conducting any work with the Cold-Seal, LM or designated personnel must provide training to his/her laboratory personnel specific to the hazards involved in working with this substance, and emergency procedures.
* The Principal Investigator must provide his/her laboratory personnel with a copy of this SOP.
* The Principal Investigator must ensure that their laboratory personnel have attended appropriate laboratory safety training and are current with any refresher training required.

I have read and understand the content of this SOP:

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