Hydrothermal Synthesis Oven
Standard Operating Procedure
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Section 1: Overview

Type of SOP: ☒Process ☐Hazardous Material ☐Hazardous Class of Materials ☒Equipment

Synopsis:
The purpose of this SOP is to provide guidelines and safety procedures for the Hydrothermal Synthesis Oven.

Section 2: Risk Assessment Summary (Hazards and control measures)

Materials:

<table>
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<tr>
<th>Material (name, CAS #, other ID)</th>
<th>Hazards</th>
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<tr>
<td>Materials used in hydrothermal syntheses in Parr and Baoshishan vessels</td>
<td>Refer to the SDS of each material used in the hydrothermal synthesis</td>
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Equipment Hazards:
Yamato oven-DKN 602C—Hot surfaces, electric shock, drive train
Parr acid digestion vessels 4744 and 4748—High temperatures, high pressures, hazardous materials (IGB)
Baoshishan Teflon lined hydrothermal synthesis autoclave reactor—High temperatures, high pressures, hazardous materials (DH)

Hazardous Conditions:
Temperature range is 10 to 260 °C. Never set the temperature out of that.

Syntheses involving fluoride media include serious hazards. An SOP for potential fluoride-based syntheses is provided separately.

Never use explosive substances, flammable substances and substances that include explosive or flammable ingredients in this unit. Explosion or fire may occur.

Do not put anything on this unit. It will cause injury if fall.

Personal Protective Equipment
Safety glasses must be worn when operating equipment for explosion and splash hazards. Nitrile gloves, closed-toe shoes and long pants must be worn. Thermal gloves should be worn when working with hot surfaces including vessels.

**Engineering Controls**

- Oven should be plugged into outlets with a ground fault circuit interrupter (GFCI).
- Oven should not be used in an area where there is a flammable or explosive gas.
- Personnel who operate the autoclave must be trained to understand proper packaging, loading, labeling, as well as operation and emergency procedures.
- Reagents used in the vessel must not react to release gas. This will lead to excessive pressure build-up.
- Before every use both the stainless steel casing and the Teflon liner must be visually inspected for cracks, pitting, rust, metal creep, or excessive wear.
- **Steel shells that are cracked or flawed in any way must be discarded.** Worn or distorted Teflon liners should be replaced.
- When charging the autoclave with reagents, the Teflon liner should never be filled more than two thirds of its maximum capacity, e.g., the maximum allowed volume in a 45-mL liner is 30 mL. This will ensure that there is sufficient headspace for vapor formation and fluid expansion when the vessel is heated.
- When working with new or unfamiliar materials it is always advisable to run preliminary experiments using small samples and observing the behavior of the reactants carefully. For best protection, the user and their supervisor should study each reaction carefully before proceeding to use the Parr digestion vessel or any other pressure vessel, asking such questions as: Is the reaction exothermic? What intermediate and final products might be produced and what will be their behavior?
- Fats, fatty acids, glycerin and similar materials must not be treated with nitric acid in these vessels to prevent the formation of materials with explosive properties.
- Nitric – sulfuric acid mixtures are not recommended for digesting organic samples due to the possibility of forming potentially unstable reaction products.
- Perchloric acid should NEVER be used in the vessel.
- Do not overheat the autoclave. The maximum temperature is 250 °C for the 45 mL vessel (Parr model 4744) and 220°C for the 50 mL – 250 mL vessel (Baoshishan).
- Do not exceed the pressure limit for each run. The maximum pressure for the 45 mL vessel (Parr model 4744) is 1800 psig and 435 psi for the 50-250 mL vessel (Baoshishan). Check relevant vapor pressure tables.
- At the end of a run, the vessel must be cooled enough to touch before attempting to remove the PTFE cup. Cooling must proceed slowly. Do not submerge the vessel in cold water as rapid cooling will tend to break the seal on the liner. An acceptable procedure is to set the vessel in a sink on an aluminum plate and run water over the plate, but not over the vessel. Cooling can be accelerated by placing the vessel in the air flow from a small fan. If it is difficult to remove the PTFE cup after a vessel has been cooled to the touch, additional cooling in a refrigerator or freezer may be necessary to shrink the cup sufficiently to remove it from the vessel body.
- Never heat a PTFE liner without slipping it into a vessel body.

A PTFE cup should be considered no longer usable and it should be replaced if it loses one percent (1%) or more of its contents when filled half- full with water and heated for thirty minutes at the intended operating temperature. Continued use of a leaky cup will expose the outer body to corrosive agents, resulting in loss of strength and possible vessel failure.
Section 3: Procedures

Installation

1. Choose a proper place for installation. Do not install the unit in a place where:
   - Rough or dirty surface.
   - Flammable gas or corrosive gas is generated.
   - Ambient temperature exceeds 35°C.
   - Ambient temperature fluctuates violently.
   - There is direct sunlight.
   - There is excessive humidity and dust.
   - There is a constant vibration.

2. Choose a correct power distribution board or receptacle. The unit electric capacity: 100 V and 14 A.

Operation mode and function list

1. Fixed temperature operation: Pressing the FIXED TEMP key enters into the fixed temperature operation setting mode. Pressing it again enters into the temperature setting mode. The "▲▼" are used to set temperature. Pressing the START/STOP key starts or stops operation.

2. Quick Auto Stop Operation: This operation is used to specify the period up to automatic stop during operation. The period up to operation stop can be set by pressing the TIMER key during fixed temperature operation. The "▲▼" are used to set the time. Pressing the START key starts the quick auto stop operation, activates the timer function and stops the operation automatically after specified period.

3. Auto Stop Operation: This operation is used to specify the automatic stop time in the fixed temperature operation. Pressing the TIMER key displays "AStp". The setting temperature "SV" can be set by pressing the ENTER key. The operation time "tim" can be set by pressing it again. Pressing the START/STOP key starts the auto stop operation.

4. Auto Start Operation: This operation is used to specify the period up to automatic start after power on. Pressing the TIMER key displays "AStr". The setting temperature "SV" can be set by pressing the ENTER key. The operation time "tim" can be set by pressing it again. Pressing the START/STOP key starts the auto start operation.

5. Program Operation: This operation is used to change the temperature according to the setting temperature and time. Pressing the PROGRAM key displays "PrG1". Press it again to select the program mode. Press the ENTER key to select the pattern "PA t". Press the ENTER key to display "End". Input the number of patterns to be used. Input the temperature and time of patterns "SV-n" and "t-n" respectively.

Note: It is impossible to change the mode during the operation. If the mode requires to be changed, stop the operation.

Tips if a problem occurs

If smoke or strange odor should come out of this unit for some reason, turn off the power key right away, and then turn off the circuit breaker and the main power. Immediately contact a service technician for inspection. If this procedure is not followed, fire or electrical shock may result. Never perform repair work yourself, since it is dangerous and not recommended.

Tips for inspection and maintenance
• Disconnect the power cable from the power source when doing an inspection or maintenance unless needed.
• Perform the daily inspection and maintenance after returning the temperature of this unit to the normal one.
• Do not disassemble this unit.
• Use a well-drained soft cloth to wipe dirt on this unit. Do not use benzene, thinner or cleanser for wiping. Do not scrub this unit. Deformation, deterioration or color change may result in.

Monthly maintenance: Check the earth leakage breaker function.

1. Connect the power cord.
2. Turn the breaker on.
3. Push the red test switch by a ballpoint pen etc.
4. If there is no problem, the earth leakage breaker will be turned off.

Standard procedure for operating Parr acid digestion vessels-model 4744 (45 mL)

This part discusses the procedure and safety guidelines for the use of Parr Teflon-lined, stainless steel autoclaves, which are used for hydrothermal syntheses in the Yamato oven.

This autoclave is composed of an internal cup and lid made of Teflon and a stainless steel casing. The instrument provides a closed system that can be loaded with reagents and used for harsh condition hydrothermal/solvothermal, high-temperature and high-pressure reactions.

Always keep the vessel upright during assembly and closing operations. Check the 277AC Bottom Disc to be sure that it is installed with the proper side facing upward to provide full diameter support for the liner. Place sample and digestion media in the PTFE cup, add the cover and slide the liner into the vessel body. Push it down as far as it will go. It may be helpful to push the bottom disc upward to meet the liner and thereby prevent air binding between the liner and disc.

Place the 286AC Corrosion Disc and the 287AC Blow-off Disc on top of the liner. Notice that two discs are required, with the thinner (corrosion) disc next to the PTFE cover and the thicker (blow-off) disc on the outside of the sandwich next to the blowout opening. Add the 241AC Spring with upper and lower pressure plates, then attach the screw cap and turn it down firmly by hand. Additional closing force applied with a hook spanner will be required to seal these vessels, but avoid over tightening. Set the vessel in the A285AC Holding Fixture and tighten the cap not more than one-eighth turn with the 264AC2 Hook Spanner. Any further tightening in excess of one-eighth turn will destroy the seal on the liner and it may put an excessive load on the screw cap, causing it to deform and possibly crack and fail.

Place the vessel in a temperature controlled oven or other heating medium and follow the proper heating and cooling procedure (see Safety Hazards). If mixing is required an optional tumbling ring (A284AC) can be attached to the vessel body. With this ring in place, the vessel will roll smoothly when placed on a powered roller, providing good agitation during long digestion procedures.

After extended use, the tapered rim on the PTFE cup will become thin and the cover may be deformed to a point where it is impossible to maintain a tight seal. When this happens, the cup and cover must be replaced. Any attempt to force a seal by over tightening the screw cap might crack the cap.

Standard procedure for operating Baoshishan Teflon lined hydrothermal synthesis autoclave reactor (50 – 250 mL)
Put the lower disc in the reactor body and make sure that it is installed with the proper side facing upward to provide full diameter support for the liner. Place sample and digestion media in the PTFE cup (max 80%), add the cover and slide the liner into the vessel body. Put the upper disc and tighten the lid. Use booster stick to tighten the lid after fully tightening with hand.

**Waste Disposal**

General safety instructions must be considered when handling the content of the autoclave after terminating reaction and opening the vessel. Wastes must be transferred to organic or inorganic waste containers accordingly.

**Section 4: Waste Disposal/Cleanup**

General safety instructions must be considered when handling the content of the autoclave after terminating reaction and opening the vessel. Wastes must be transferred to organic or inorganic waste containers accordingly.

**Section 5: Emergency Response**

**Power is lost**
- Turn off oven
- Turn off rotating motor (IGB)
- Call F&S
- Call Ohsung (217-418-4871)

**Parr/Baoshishan vessel burst**
- Call Ohsung (217-418-4871)

**Liquid or fume leaking from the vessels out of the oven**
- Turn off oven
- Turn off rotating motor (IGB)
- Inform everyone to move to safety
- Call DRS
- Call Ohsung (217-418-4871)
Training Documentation
Signing this document means that you have read and understand all aspects of this Standard Operating Procedure. The supervisor is the person that acknowledges you took the training and understand the procedure. They can be a lab manager or researcher assigned by the PI to oversee this particular SOP.

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