CEAS Laboratory Standard Operating Procedure (SOP)

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| Procedure Title | | **Operation of Autoclave (Piston) in 551 Mantei Center** | |
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| Date of  creation / revision | | Date created: 8/10/2024 | Date revised: 10/4/2024 |
| Principal Investigator | | Gregory Beaucage | |
| Location  (Building and room number) | | Mantei Center 551 | |
| 1. | | **This standard operating procedure (SOP) is for:** | |
| Use of specific chemical or class of chemicals with similar hazards  Butane  Specific laboratory procedure or experiment  Use of specific lab equipment  Operation of the autoclave | | | |
| 2. | | **Process or experiment description**  *Briefly summarize the process or experiment, including volume and concentration of chemicals, how long each experiment or procedure runs, and how frequently it will be conducted.* | |
| The autoclave is used to dissolve and separate plastic waste from additives and to separate various plastic components in the waste stream. | | | |
| 3. | | **Hazard and risk assessment**  *Identify and list ALL potential hazards including chemicals, equipment, and other physical hazards. For chemical hazards, list the GHS hazard class and rating and OSHA occupational exposure limits. Attach safety data sheets (See section 12). The UC SDS database links can be found on* [*https://www.uc.edu/about/admin-finance/ehs/resources-and-documents/advisories.html*](https://www.uc.edu/about/admin-finance/ehs/resources-and-documents/advisories.html)*.* | |
| **Chemical Hazards:**  Butane (80 ml draw from a can of liquid butane 250ml at 0.2 MPa and room temperature) a trace amount of mercaptan is included in the butane to aid leak detection. (SDS attached)    Dry Ice. The autoclave is cooled with dry ice which is placed on the top of the autoclave to help maintain liquid butane. (SDS attached)  The process involves dissolving mixed plastic waste containing polyethylene, polypropylene, polystyrene, PET, paper, adhesive, ink, titania, silica, carbon black fillers. This mimics the residential recycle stream. The process does not involve any chemical reactions and these materials remain inert through the process. The process just separates these household plastics.  **Physical Hazards:**  Liquid butane is heated to 150°C in the autoclave and reaches a pressure of about 50 MPa. The autoclave has been tested to 100 MPa by the manufacturer. The device has two blowout valves that will release the pressure at 80 MPa. The operating procedure below, involves venting the autoclave at pressure which is the same as having one of the blowout valves being released at 80 MPa.  The autoclave is milled from a single piece of stainless steel 3 inches thick with a 1-inch stainless steel lid held on with 8 bolts. It is possible that the Teflon seal between the autoclave body and the lid could leak but that would occur during heating or filling and would result in a small leak and depressurization. The system wouldn’t pressurize if the Teflon seal leaked.  There is a possibility that the quartz window (2 mm diameter disk) at the base of the autoclave could crack. The window is viewed using a fiberoptic camera that fits in the window hole. This would lead to a slow leak and depressurization.  Fire: butane is flammable at concentrations between 1.8 and 8.5% in air. The autoclave works with liquid butane with is not flammable. In the hood leaks are quickly diluted to below 1.8%. There is a narrow opportunity in concentration where a flame could ignite as the butane is diluted in the hood. There are two flammable gas detectors, a hand held and a fixed detector that will alarm when a flammable concentration is present.  Butane gas is heavier than air so the hood is set appropriately.  Burn hazard due to the operating temperature of 150°C. A “Hot” sign will be used and heat gloves will be used to operate the autoclave. | | | |
| 4. | | **Safety equipment**  *Specify all equipment needed to perform the procedure safely and to respond to emergencies.* | |
| 4.a. | Engineering / ventilation controls  Work will be done in fume hood with baffles set for use of heavier than air gas  Butane/Propane detectors (one in hood, one handheld for leak detection)  Details on the Hood and butane removal:  Entrance width 126cm  Entrance height when venting 30cm  Maximum entrance height 68cm  Inner depth 60cm  Inner height 120cm  The ventilation performance of the hood we use is 100 fpm, and it is a variable air velocity (VAV) hood that always maintains 100 fpm depending on the area of the hood window opening.  First, the volume inside the hood is 907,200 cm^3.  When all the liquid butane of the current piston is discharged, the expanded gas volume is 17,947 cm^3 at atmospheric pressure and room temperature.  (For a new larger piston not part of this SOP when all the liquid butane of the piston being designed is discharged, the expanded gas volume is 74,776 cm^3.)  Since the internal volume is about 50 times larger than the existing one and about 12 times larger than the later one, there is no leakage at all when the hood is closed.  The air ventilation speed inside varies depending on the degree of window opening.  When venting, since the experimenter opens the valve by hand, opening the window about 30 cm will not interfere with the movement of the arm, secure a field of vision, and properly protect the experimenter's respiratory system.  Under this partly opened condition, the volume of gas vented per second according to the window open face is 192,024 cm^3.  In the case of the current piston, the vented butane would be completely vented in 0.09 seconds.  (In the case of the future piston, the vented butane is completely vented in 0.39 seconds.)  We can conclude that it is a very safe condition. | | |
| 4.b. | Personal protective equipment  Eye protection glasses  Nitrile chemical gloves  Heat resistant gloves    Fire resistant lab coat | | |
| 4.c. | Location of nearest emergency safety equipment | | |
| Item | | Location | |
| Eyewash / safety shower | | 551 Mantei Center | |
| First aid kit | | 551 Mantei Center under sink | |
| Spill kit | | Chemical spill kit under sink, Oil spill kit under hood | |
| Fire extinguisher | | 551 Mantei Center | |
| Fire alarm manual pull station | | Mantei Center near the freight elevator | |
| Telephone | | Ordered | |
| Other | | N/A | |
| 5. | | **Step-by-step methodology**  *Provide a sequential (Step 1, Step 2, Step 3, etc.), detailed description of procedure or experiment and when special safety equipment and safety precautions are to be utilized. Include temperature, pressure, and other conditions required in the experiment. Include schematics, diagrams and/or photos for complex setups.* | |
| **Pre-operation Inspection:**  Check for proper arrangement of the autoclave  Secure butane tank with chemical stand  Verify installation of blowout values  Make a note of the use time, personnel, and protocol to be followed in the instrumental log book  Wear PPE as described in Section 4.b.  **Operational sequence:**     1. b) c)     d) e) f) g)    h) i) j) k)    l) m)  Figure 1. a) Autoclave with check tube at back for filling with liquid butane. b) placing dry ice on top of autoclave to cool autoclave ensuring liquid butane fill. c) Manual for fixed combustable gas detector. d) Manual for hand held combustable gas detector. e) Autoclave with lid removed showing Teflon seal. f) Video from fiber optic through autoclave window. g) Butane canister attached to autoclave for filling. h) Closeup of filling autoclave with liquid butane. i) Vent tube removed from autoclave lid. j) sealing lid to autoclave. k) Venting autoclave, l) Showing audoclave with attached lid and thermostat. m) Two blow-out valves vertically aligned and tethered to a >10kg weight.   1. Remove the top vent valve assembly including check tube from the autoclave lid (Fig. 1 i, j) 2. Open lid to autoclave, place plastic pieces and magnetic stirrer in the autoclave (Fig. 1 j, e ) 3. Close and secure the autoclave lid with the eight bolts tightening opposite sides of the lid in sequence to ensure an even seal (Fig. 1 j) 4. Attach the top vent valve assembly to the autoclave lid (Fig. 1 a, h) 5. Connect a full liquid butane can to the autoclave (Fig. 1 g, h) using the quick connect connector.   Flammable gas that is heavier than air  Work only with small canisters of butane and propane (<100ml)  Extra canisters are stored in the flammables cabinet  Only the maximum amount of alkane needed for the experiment will be present in the hood  (typically two canisters)  Empty canisters should be clearly marked empty and placed in hazardous waste  Hood baffles must be set to the marking for heavier than air gas  (lever on right side of hood). If other work is done in the hood the baffles need to be set  appropriately for the type of gas being exhausted.   1. Cool the autoclave using dry ice (Fig. 1 b) 2. Close the vent valve, then fill autoclave with liquid by opening the valve to the supply can while slowly opening and closing the vent valve while monitoring pressure (Fig. 1 g, h) so that about 0.2 MPa is maintained in the autoclave.   Check release valve tube (Fig. 1 a, b) which will fill with liquid when the autoclave is filled with liquid  since it is the highest point in the autoclave.  8) Close exit valve to autoclave  9) Close feed valve to autoclave  10) Vent the feed tube  11) Remove the butane supply canister, mark the amount used on the canister, return canister to  the flammables cabinet  12) Heat autoclave to desired temperature for supercritical fluid while monitoring the pressure using the  autoclave temperature controller (Fig. 1 a, b and l, black square to right front of autoclave) and an  external thermocouple in contact with the autoclave lid (the lid temperature significantly lags the  autoclave thermostat). The temperature controller is adjusted by changing the setpoint. Do not set the   temperature above 180°C as written in magic marker near the temperature controller.  Heat resistant gloves should be used at temperatures above 40°C (100°F)  The pressure on the gauge should not exceed 60 MPa. If the pressure exceeds 60 MPa then drop the   setpoint to room temperature, unplug the temperature controller and manually vent the system to below  60 MPa.  **Place HOT sign clearly visible near the autoclave when it is above 40°C**  13) Perform thermal cycle that is desired, typically  Remain at temperature for 10 minutes to 4 hours  Monitor the pressure to make sure that the pressure does not exceed 60 MPa. If it exceeds 60 MPa  discontinue heating and manually vent the system to 60 MPa as in step 12.  Cool to crystallization temperature typically 100°C while monitoring pressure  Crystallize for desired time, typically 10 minutes to 2 hours  Depressurize the system by manually venting the alkane  Open the system to remove the product  **The operation of the autoclave is limited to a maximum temperature of 180°C and a maximum**  **pressure of 60 MPa.**  **Post operational tasks:**  Clean the autoclave  Remove the vent valve assembly  Remove the lid  Remove the polymer precipitate and store the product in a vial  Clean the autoclave chamber with a paper towel to remove residual plastic components  Clean the stir bar by washing  Clean the vent valve and blow air through the vent valve  Reassemble the autoclave  Store and label the samples, make notes in instrumental log book  **In the event of a gas leak:**  In the event of a gas leak secure the device and ensure that the leak is removed. The device is  secured by turning off the heat/power and venting the butane from the top release valve being  careful of hot liquid spray that may result.  **In the event of a blow out value rupture:**  The autoclave operates at medium pressure up to 80 MPa  Device has two blowout valves set at 80 MPa. The release from these valves is identical to the   normal release from the exhaust valve at the top of the autoclave. The autoclave weighs about 30  kg (60lbs) so the release of a few ml of liquid butane at 80MPa does not have sufficient force to  move the autoclave. Also, the shaking of the blowout valve and connected tube is completely canceled out by the 10kg fixed bracket.  Pressure is manually monitored during heating  In case of unexpected over pressurization:  Turn off the heat and open release valve  Use hot gloves to protect from hot vapors | | | |
| 6. | | **Designated area**  *Where highly toxic, highly reactive/unstable, highly flammable, or corrosive materials are used, identify the designated work area(s) and the necessary decontamination after completion of work. For more information, see the* [*Generic Chemical Hygiene Plan for the University of Cincinnati*](https://www.uc.edu/content/dam/refresh/af-62/ehs/forms/UC-CHP.pdf) *and* [*UC EH&S Advisories*](https://www.uc.edu/about/admin-finance/ehs/resources-and-documents/advisories.html)*.* | |
| Work is done in the hood in 551 Mantei Center | | | |
| 7. | | **Special handling procedures, transport, and storage requirements**  *Describe special handling and storage requirements for hazardous chemicals used in this procedure, especially those that are highly reactive/unstable, flammable, toxic, or corrosive. Describe secondary containment requirements for transport between laboratory rooms. For more information, see the* [*Generic Chemical Hygiene Plan for the University of Cincinnati*](https://www.uc.edu/content/dam/refresh/af-62/ehs/forms/UC-CHP.pdf)*,* [*UC EH&S Advisories*](https://www.uc.edu/about/admin-finance/ehs/resources-and-documents/advisories.html)*, the* [*UC Office of Biosafety*](https://research.uc.edu/support/offices/biosafety/)*, and* [*UC Office of Radiation Safety*](https://research.uc.edu/support/offices/radsafety/overview#:~:text=The%20mission%20of%20the%20University,used%20under%20the%20University's%20Radiation)*.* | |
| Flammables are stored in the flammables Cabnet | | | |
| 8. | | **Waste disposal**  *Identify and list all hazardous waste to be generated and appropriate disposal procedures. Include liquid and solid waste.* | |
| No hazardous waste is generated. Process operates on domestic garbage.  The process does not involve chemical reactions, it is a physical separation of different grades of plastic and settling out of additives and fillers, so the waste generated is the same material that was put into the autoclave, mixed plastics waste such as flexible packaging, water bottles, polyethylene and polypropylene.  Request chemical waste pick-up online at: https://ehs.uc.edu/Chemical/ | | | |
| 9. | | **Emergency procedures**  *Describe how spills, chemical exposure, and other accidents should be handled and by whom. List emergency contact numbers. More information on emergency procedures can be found in the* [*Generic Chemical Hygiene Plan for the University of Cincinnati*](https://www.uc.edu/content/dam/refresh/af-62/ehs/forms/UC-CHP.pdf)*,* [*UC Emergency Management website*](https://www.uc.edu/about/publicsafety/emergencymanagement.html)*, and* [*UC EH&S Emergency Procedure section of their website*](https://www.uc.edu/about/admin-finance/ehs/emergency-procedures.html)*.* | |
| **(REVIEW AND MODIFY THIS SECTION AS NEEDED TO ENSURE THAT THE FOLLOWING EMERGENCY PROCEDURES APPLY. DELETE THIS RED TEXT WHEN FINISHED.)**  **Emergency Procedures**   * Dial 911 for: * Fires * Toxic Gases * Serious Injuries * Give the operator:   + The exact location (building and room number)   + Type of emergency * Assistance needed   **1. Fires**   * Call 911 immediately. * Notify others in the area. * If the fire is large, evacuate the area. Pull the fire alarm (alarms are located in the hallways near the exits). * If the fire is small and you are confident that you can extinguish it safely, you may attempt to do so, but only after others have been informed and 911 has been called.   **2. Chemical accidents**  a. Chemicals in the Eye: If you get chemicals in your eyes, what you do in the first few seconds is the most important:   * Yell for help; you will need it! * Get to an eyewash station or other source of water. * Flush with water for at least 15 minutes. Use thumb and forefinger to hold eyelids open. * Do not rub your eyes or put medication in them. * Do not put chemical neutralizers in your eyes. * Have someone call 911. * Always report to the University Health Services after flushing with water for 15 minutes. All accidents must be reported.   b. Chemical Splashes on Your Person: Wash thoroughly with water.  Depending on the extent of the contamination and hazard of the chemical, you may need to:   * Yell for help! * Run to the safety shower! (A sink may be used if more appropriate.) * Have someone call 911 (emergency). * Remove all contaminated clothing. * Wash thoroughly with water; 15 minutes is recommended. * Report to the University Health Service after washing. All injuries must be reported.   c. Inhalation of Chemicals: If a person has been exposed by inhalation:   * DO NOT become a victim yourself! If more than one person is down, a hazardous situation exists. DO NOT enter the room. * Evacuate the area and dial 911 (emergency). Even one person down might indicate the presence of toxic gases. Call 911 and proceed only with extreme caution. * Get the victim to fresh air if you can do so safely. * Apply artificial respiration as appropriate if you are properly trained to do so. * Call 911 (emergency).   **IMPORTANT: ALL SPILLS:**   1. **Evacuate the laboratory at once if you are not certain the situation is non-hazardous, and call Environmental Health and Safety at 556-4968. If you KNOW the situation is non-hazardous, there is no need to evacuate the laboratory.** 2. **If you have any doubt about your ability to safely clean up the spill yourself, call 556-4968 to enlist the aid of Environmental Health and Safety. After consulting with Environmental Health and Safety, it may be determined that it is safe for you to clean up the spill, or it may be determined that it is appropriate for Environmental Health and Safety/Hazmat team to clean it up.** 3. **Hazards of a spill to consider:**  * **Are there toxic vapors being given off that may be harmful to breathe?** * **Is it a flammable solvent that might burst into flame at any moment?** * **Are there sources of ignition such as a hot plate or vacuum pump nearby?** * **Could it be toxic if it got on your skin?** * **Is it an acid or base that could burn your skin or eyes?** * **Is it inside or outside a hood (Spills in laboratory fume hoods are much less hazardous)?**   **Note: solvents, water, and especially hydroxide solutions make a floor very slippery.**  **Building maintenance emergencies** (for example: power outages, plumbing leaks, fume hood malfunction)  Call (513) 558-2500 and after-hours call (513) 556-1111 to report a facility emergency.  **Incident Reporting**  Any injury to a University employee, student, or visitor must be reported within **twenty-four (24) hours after occurrence**. Complete an INITIAL REPORT ON WORK-RELATED INJURY or ILLNESS online or offline (PDF download) at the [EH&S Work-related Injuries & Illnesses section of their website](https://www.uc.edu/about/admin-finance/ehs/emergency-procedures/work-related-accidents-injuries-illnesses.html) (www.uc.edu/about/admin-finance/ehs/emergency-procedures/work-related-accidents-injuries-illnesses.html).  ***Additional information for UC employees:***  An employee who has an occupational/work-related injury or illness must report the incident to his/her/their supervisor as soon as possible, but no later than 24 hours after the occurrence.  Medical Treatment   * If medical treatment is necessary for work-related injuries or illnesses, the employee should obtain treatment at the following locations: * UC Health, Employee Health & Wellness Clinic (UCH), 3200 Burnet Avenue, South Entrance. Phone: 585-6600. Monday through Friday, 7:30 am to 4:00 pm. * All Other Times and Holidays: University Hospital, Inc., Center for Emergency Care, 234 Goodman Avenue. Phone: 584-5700. * To ensure proper processing of Workers’ Compensation claims, an employee must tell the provider or medical facility that the medical treatment is for an occupational injury or illness. The injured employee must file a BWC claim for payment of charges when medical treatment is obtained from a provider other than University Health Services. * Before obtaining further medical services, the employee must coordinate follow-up treatment with University Human Resources by calling 556-6381. | | | |
| 10. | | **Training requirements**  *List the general and laboratory-specific training required for authorized users of this SOP.* | |
| 10.a | EH&S OSHA Hazard Communication Training  EH&S EPA Hazardous Waste Training  EH&S OSHA Bloodborne Pathogen Training  Radiation Safety Training (<https://research.uc.edu/support/offices/radsafety>)  Other: Review of chemical safety data sheet(s)  Other: Demonstrate proficiency of step-by-step methodology including safety procedures  Other: Demonstrate proficiency on executing emergency response procedures | | |
| 10.b | **Additional training requirements**  *A complete list of UC EH&S training can be found at* [*https://www.uc.edu/about/admin-finance/ehs/training.html*](https://www.uc.edu/about/admin-finance/ehs/training.html) *and Fire Extinguisher Training is available from UC Public safety at* [*https://www.uc.edu/about/publicsafety/fire/fireextinguishertraining.html*](https://www.uc.edu/about/publicsafety/fire/fireextinguishertraining.html)*.*  N/A | | |
| 10.c | **Additional prior approvals required**  *List any tasks that require prior approval by the principal investigator (for example, working outside of normal business hours, use of restricted chemical and other higher hazard chemicals, running of higher hazard operations, or any deviation to this SOP):*   1. Working outside of normal business hours, 2. Use of restricted chemical and other higher hazard chemicals, 3. Running of higher hazard operations, 4. Any deviation to this SOP | | |
| 11. | | **Safety References and other Attachments**  *List books, published papers, equipment safety manuals, webpages, and any other references used in writing this SOP. Attach chemical safety data sheets, schematic diagrams, and photographs for complex procedures.* | |
| Shianjia Equipment Company designed and built the autoclave. www.Shianjia.com  A printout from their webpage is attached.  SDS for butane and dry ice is attached to this SOP  <https://www.globalp.com/wp-content/uploads/2019/10/SDS_Butane_Final.pdf>  <https://www.airgas.com/msds/001091.pdf>  The process is intended to use standard household plastic recycle which includes polyethylene, polypropylene, PET, flexible packaging (multilayer films). These are food grade materials so have no known hazards. There is no chemical reaction, so the materials are unchanged by dissolving them and precipitating them in butane. There are no hazards involved with the process. MSDS for polyethylene, polypropylene, PET are included.  <https://korellis.com/wp-content/uploads/2016/01/POLYETHYLENE.pdf>  <https://advancedpetrochem.com/sites/default/files/MSDS%20Advanced-PP%20Homopolymer%20Updated%20April%202016.pdf>  https://www.chemos.de/import/data/msds/GB\_en/25038-59-9-A0017291-GB-en.pdf  Original design drawings for autoclave (lid was modified to a simple plate with a vent)    The autoclave consists of a cylindrical chamber cut from a block of stainless steel with 3-inch walls and a lid that is 1-inch stainless steel held by four bolds and a teflon seal. The autoclave has cartridge heaters and a temperature controller. There is a magnetic stirrer on the bottom of the device. The autoclave chamber is a cylindrical cavity 1 inch in radius and two inches high with a volume of 100 ml. The chamber has a 1-inch-thick quartz window for a CCD camera connected to a fiber optic that is ¼ inch diameter. There is a single inlet to the piston that is 1/8-inch stainless steel tubing with swagelok fittings and an outlet valve that is also a 1/8-inch stainless steel connection. Two blow out valves at 80 MPa are attached to the inlet and to the outlet lines. The liquid butane feed uses a standard quick connect for refilling butane lighters. There are two back flow preventers along the feed line, one in the standard butane quick connect and a second along the line to the autoclave. | | | |