

Standard Operating Procedure for Soxhlet Extraction of Biomass

Ragauskas Group Safety Meeting

Soxhlet Extraction of Biomass

- Used for the removal of soluble materials or extractives from wood, pulp, grasses, etc.
- Extractives include non-structural components of biomass samples which potentially could interfere with the analysis of the sample and thus must be removed prior to compositional analysis.

Solvent Systems Used in Soxhlet Extraction of Biomass

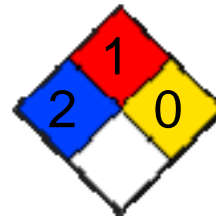
- Dichloromethane
 - Used to remove waxes, fats, resins, photosterols and non-volatile hydrocarbons
- Ethanol-Benzene (1:2 v/v)
 - Removes above substances in addition to low-molecular weight carbohydrates, salts and tannins

Potential Hazards

- Chemical
 - Possible exposure to hazardous solvents

Dichloromethane

- Carcinogen



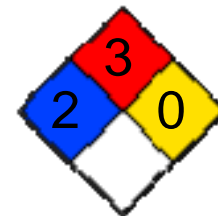
Ethanol

- Flammable



Benzene

- Carcinogen
- Mutagen
- Flammable



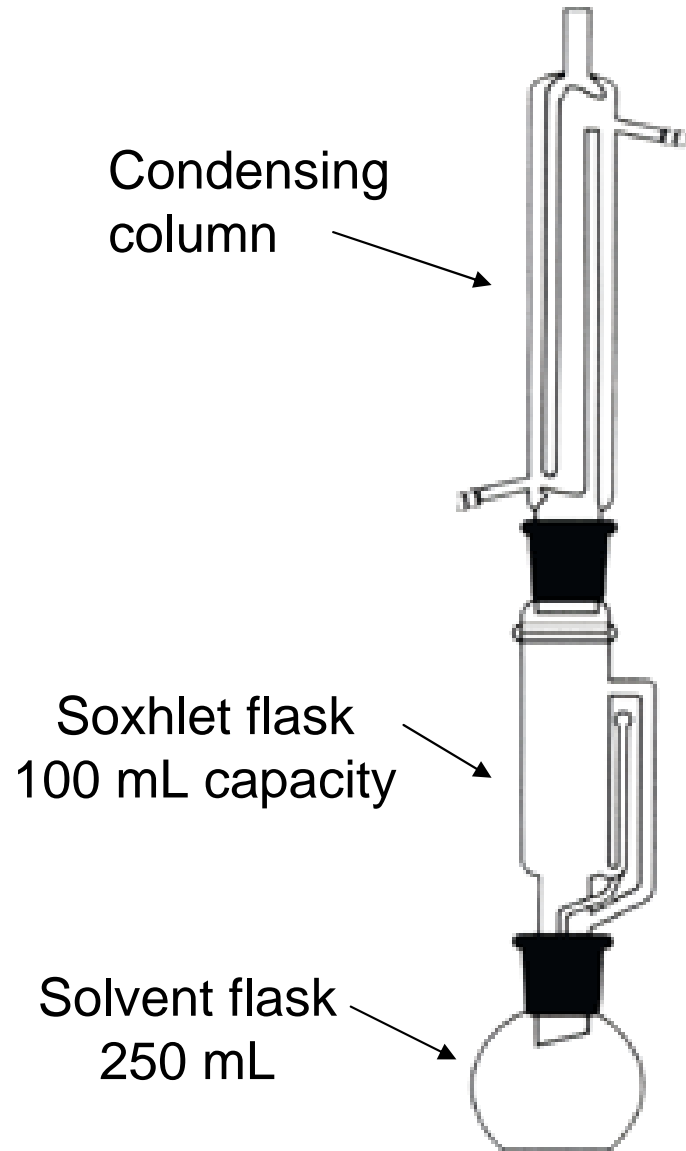
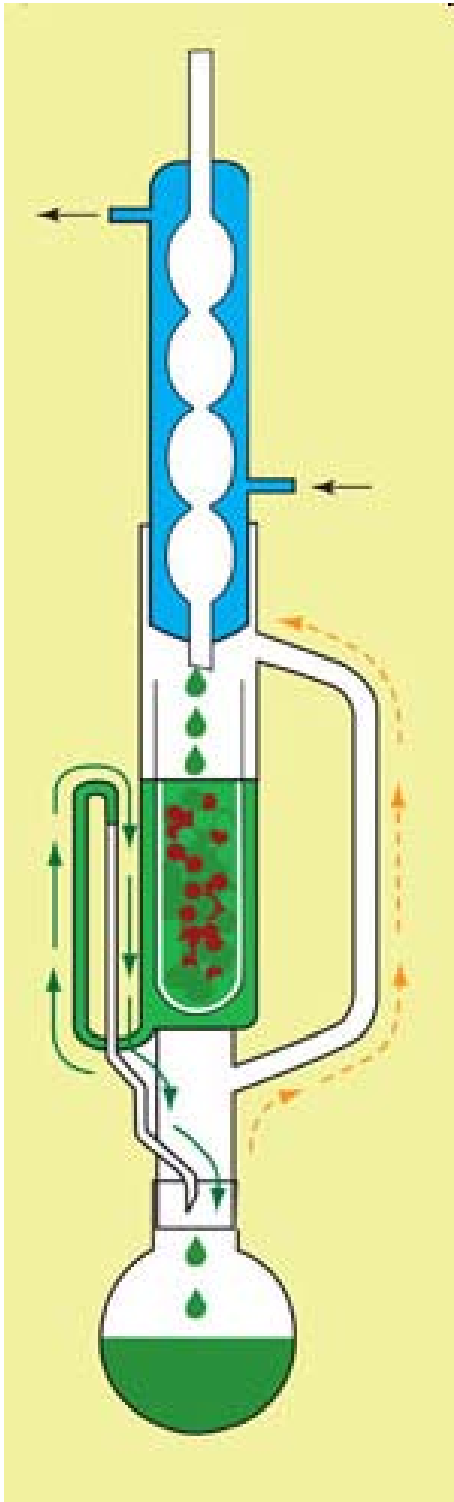
Potential Hazards

- Electrical
 - The heating source is electric. Never touch exposed wires, make sure system is grounded and use GFI outlets when possible.
- Physical
 - Refluxing solvents and glassware are HOT. Use caution when monitoring extraction and breaking down apparatus to avoid heat burns.
- Fire
 - Solvents used are flammable.

Safety Precautions

- Fume Hood
 - Always perform Soxhlet extractions in a fume hood (with sash down) to avoid inhalation of hazardous solvents and to contain potential fire
- Personal Protective Equipment
 - Eye protection: required
 - Hand protection: required
 - Nitrile gloves are recommended
 - Insulated gloves used for contact with hot glassware
 - Protective Clothing: lab coats, closed toe shoes and long pants should be worn

Soxhlet Apparatus



Extraction Thimble



Heat Source



Boiling Chips



Soxhlet Procedure



- 1) Hookup the water lines to the condenser. Be sure to secure the lines with copper wire or plastic straps to avoid water spillage. The top of the condenser should be open to the fume hood. DO NOT CAP.
- 2) Place the specimen into a tared cellulose extraction thimble. Be sure to leave approx. 1 cm space between sample and top of thimble. Cover the top of the thimble with filter paper or Kimwipe.
- 3) Place the thimble in the Soxhlet flask by sliding it in with the flask held sideways and then secure the Soxhlet flask to the condenser.
- 4) Place several boiling chips into a round bottom solvent flask.
- 5) Fill the solvent flask with no more than 60-70% of the desired solvent or solvent system.
- 6) Attach the solvent flask to the Soxhlet flask.
- 7) Turn on the water flow to the condenser, must have a water detector in the fumehood.

Soxhlet Procedure



- 8) Turn on the water flow to the condenser, post a completed operating equipment notice posted (see pg 12).
- 9) Place the round bottom solvent flask into the heating source. If using a large heating mantle, apply glass fiber to the sides and top of the flask to reduce heat loss.
- 10) Adjust the heater to provide a boiling rate which will cycle the specimens approximately every 10-12 minutes.
- 11) Allow the extraction to run for 24 hours or approximately 120 cycles.
- 12) Place an operator placard on the fume hood during operation
- 13) At the end of the last extraction cycle turn off the heat source and remove the Soxhlet flask from the setup.
- 14) Remove the extraction thimble and allow to air dry in the fume hood

Emergency Shutdown Procedure

- In the event of a water line break, glassware problem or solvent leak immediately shut off the power to the heat source, turn off the water and close the hood sash.
- Once the system has cooled, contain any spills and proceed with normal cleanup and disposal procedures.

Cleanup and Disposal

- If the extractive composition is to be analyzed, remove most of the solvent from the solvent flask via rotary evaporation and then transfer contents to an aluminum dish and place in fume hood to air dry.
- If not, then place the liquid contents of the solvent flask into an appropriate organic waste bottle for disposal.

Operating Equipment Notice

N O T I C E

OPERATING EQUIPMENT

This equipment is being used and was left in operation by:

NAME _____ DEPARTMENT _____

POTENTIAL HAZARD _____

IN AN EMERGENCY, CONTACT

PRIMARY _____ ALTERNATE _____

Office Room No. _____ Office Room No. _____

Office Phone No. _____ Office Phone No. _____

Home Phone No. _____ Home Phone No. _____

PROCEDURE TO TURN OFF THIS EQUIPMENT
