Standard Operating Procedure
for
Hazardous Chemicals

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Building and rooms: Life science bldg, Lab B 310

Chemical(s)	Ethyl Alcohol 190 proof
Process	**Soxhlet Extraction**
Specific Hazards	Ethyl Alcohol is flammable, and may be harmful upon skin/eye contact or ingestion.
Personal protective equipment	: 3-5 mil nitrile gloves : lab coat : chemical safety goggles (when splash potential exists)
Engineering/ventilation controls	: chemical fume hood : emergency shower and eyewash accessible
Special handling procedures and storage requirements	Stored in common container by hood in B310.
Spill and accident procedures	Eye exposure: Wash eyes for at least 15 minutes, lifting the upper and lower eyelids. Seek medical attention immediately. Skin exposure: Rinse affected skin with plenty of water while removing contaminated clothing and shoes. Rinse for at least 15 minutes. Seek medical attention.
	Small (< 2L): Extinguish all sources of ignition. Dilute with water and mop up or absorb with spill pads. Notify PIs.
	Large (> 2L): Extinguish all sources of ignition, notify PIs and absorb with DRY earth, sand or other non-combustible material. Do not touch spilled material.
Waste disposal	Waste should be collected in a properly labeled hazardous waste container.
Special approval	No special authorization needed after SOP training and reading MSDS.
Prepared by	Updated by Kavita Aulakh/Scott Harding, 03/24/17
Reviewed by	Name/date:

Soxhlet Extraction

Chemicals:
Ethyl Alcohol 190 proof

Tea Bags and heat-sealer (to seal tea bags filled with samples)

Recommended program:
- EtOH

Cycling settings
- Cycles 99
- Heating 120%
- Time 0

Procedures:

EXTRACTION
*To do extraction for just 1 chamber you need at least 110mL but no more than 150mL. For all 6 chambers you need at least 1L.

1. Prepare your samples by sealing them in tea bags (1/2-3/4 full per tea bag, **don't overfill**). Use lead pencil to label your tea bags (ink will be washed away with ethanol).
   Consider your downstream analysis need. You may need >500 mg for comprehensive wood chemistry or just <20 mg for MBMS.
2. Soak tea-bagged samples in 190 proof ethanol for 2-3 minutes to saturate them.
3. Use 4 (3/4 full) tea bags per column. More bags can be added if samples are small and bags were heat-sealed and trimmed accordingly.
4. Make sure the hood is down and pull out the extraction chambers. The unit should be clean and dry.
5. Using blunt tongs, load tea bags into one of the glass cylinders stored in the rack behind the Soxhlet unit. Be sure the tissue powder is toward the bottom.
6. Place each cylinder with samples into one of the extraction chambers.
7. Adjust the optical detectors height with the knob above the extraction chambers.
8. Push the chamber in and lift the hood.
9. Add 3 boiling stones (If you add more/less, make sure you put the same number of stones in each solvent boiling beaker).
10. Pour ethanol in the boiling beaker and fill just **above** the Buchi mark, not just below).
11. Put the solvent beaker back in and put hood down.
12. Make sure cold water is running into the condensation coils (the extraction until will not start if there is no/not enough water running into the condensation coils).
13. Press the "Down" button on the keypad.
14. Check the program and change in "File" if needed, or program in a new program and save.
15. Press "Start" on the keypad.
16. When the switch opens, level of solvent should be above of the Buchi logo.
17. **IMPORTANT!!** Check ethanol level after initial 2-3 cycles and add more ethanol from top as needed.
18. After extraction is complete, remove your solid samples and the dried analytes in the solvent beaker by pressing the "Up" button on the interface. Lift the hood to reach the solvent beaker. Make sure the hood is down and pull out the extraction chamber to reach your solid samples.

*Note: If the solvent gets low/starts to run out, a warning will come up and pause the cycles until more solvent is added. Solvent can be added with a funnel into the top of the condensation apparatus.

*"Abort" stops the whole program. Water will run in the condenser until the hot plates are below a specific temperature.

*"Skip" will go to next stage (i.e. if the instrument is in the extraction stage, skip will take you to the rinse stage).

WASTE DISPOSAL
Waste should be collected in a beaker and then transferred to the appropriate hazardous waste container

MENU FUNCTIONS (**not used very often)

1. Occupied position – Choose which extraction chambers will be used.
2. File – can select which program to use
    a. go to open and select your program using the turn knob
3. Program – can program the parameters for the solvent you plan on using or select from already programmed solvents. This can be saved and used for later extractions for similar/same type of samples and solvents
    a. Preprogrammed solvents – has the appropriate parameters for a few solvents.
        i. Chloroform
        ii. Hexane
        iii. Petroleum Ether
        iv. Diethyl Ether
    b. Custom – allows you to set parameters for solvents that are not preprogrammed.
        i. Extraction Stage – can set the time of run, number of cycles to run, or both. If both the time and number of cycles is set, the extraction stage will continue until both criteria are met. You can also set the heating temperature (in power percentage).
        ii. Rinse Stage – the valve is open in this stage so the solvent continually moves through the chamber. This stage doesn't have to be many hours long. You can set the time and heating temperature.
        iii. Drying Stage – This stage separates the analytes from the solvent. The solvent is heated into a gas stage and condenses into the solvent tank. You can set the time and heating temperature.

    *When choosing temperatures for use with a mixed solvent, select the temperature based on the solvent with the highest boiling point.
    *To save the program: go to "File" after parameters are set → "Save" → "Name"

4. ** Instrument Settings – These show the instrument parameters set at installation.
5. **Service Functions – This function is used for troubleshooting problems and checking firmware version installed. You can check temperatures, valves, sensors, operating hours, and unit information.
6. ** Mode – has 2 sub-functions
    a. Service – only used with service engineers for problems with the instrument.
    b. Extraction – most common mode and is used by analysts to use the instrument

HOW IT WORKS:
The extraction process is a series of cycles. The solvent is heated and converted into a gas, the gas rises into the condensing coils where it cools down and converts back into a liquid, and liquid solvent drips into the extraction chamber. A closed valve keeps the solvent in the extraction container until a set level is reached, the valve opens and the solvent drains into the solvent beaker taking the leached analytes, and the next cycle can begin.

The analytes being leached from the solids are typically less volatile that the solvent and are unlikely to heat enough to turn into a gas and go into the extraction chamber with the solvent. This means that the solvent going into the extracting chambers after each cycle is still pure solvent.

*Please note that this is not a closed system so if the water isn't cold enough, gas can escape through the holes in the top