Glassware Cleaning and Muffle Furnace Protocols in the Organic Preparations Laboratory

FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION

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1. SCOPE AND APPLICATION

1.1. The techniques described in this technical procedure are used to prepare glassware and other sample handling devices for semi-volatile organic and pesticide analysis. The basic objective is to remove all traces of organic compounds of interest and interferences from surfaces that may come in contact with a sample. Criteria for acceptance of clean bottles for sample collection are also described.

1.2. The cleaning procedures are designed for specific laboratory items that come into direct or indirect contact with the environmental or quality control samples. The procedures must be followed strictly in order to minimize contamination risks to the samples and instruments. The concepts and techniques contained within this Standard Operating Procedure (SOP) may also be applicable for cleaning other types of equipment for organic analysis, but caution must be exercised. Verification of equipment cleanliness though the use of method blanks and quality control checks are also described.

1.3. The cleaning and maintenance schedule for the individual analytical instruments is not discussed in this SOP, but is described in the operator manual for each instrument. References for the analytical instruments are found in the analytical method SOPs.

2. METHOD SUMMARY

The cleaning of glassware is a multi-step process where glassware is initially cleaned by a general cleaning procedure and then further cleaned for specific tasks. It is imperative that glassware and other sampling handling utensils be cleaned properly and safely prior to sample preparation.

The use of a muffle furnace to remove trace levels of organics is a commonly used technique in the laboratory. Appropriate safety procedures, personal protection equipment and protective clothing must be employed at all times.

Only trained personnel are allowed to use the muffle furnace.

3. APPARATUS AND EQUIPMENT

3.1. Type-2 DI Water System– The laboratory is equipped with an in-house DI water system for general cleaning purposes.
3.2. Type-1 Aqua Solution 2000 Water Purification System – The laboratory has 18 mohm Type-1 DI water available in the Volatiles laboratory for situations requiring a high-grade DI water.
3.3. Fisher IsoTemp Forced-Air Oven

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3.4. Wilt Model 210 Muffle Furnace
3.5. Lancer Model 1400 Laboratory Dishwasher
3.6. Brushes:
  3.6.1. Large Beaker Brush
  3.6.2. Test Tube Brush
  3.6.3. Nessler Tube Brush
  3.6.4. Narrow Tube/Pipet Brush
3.7. Gloves:
  3.7.1. Heavy Duty Latex Gloves
  3.7.2. Neoprene Gloves
  3.7.3. North Silver Shield Gloves
  3.7.4. Autoclave/Oven Gloves
3.8. Aluminum Foil, heavy-duty
3.9. Pressurized Air and Regulator
3.10. Pressured Nitrogen Gas and Regulator
3.11. Oil-Free Vacuum Pump and Traps
3.12. Fisher FS14 Ultrasonic Cleaner with basket
3.13. Kappler Tyvek Lab Coats – The Tyvek coats are used for cleaning since they are disposable, chemical resistant and water resistant.
3.14. Technicloth Wipes or Kimwipes – lint-free cloth wipes
3.15. Standard Folded Paper Towels
3.16. Ziplock Bags, Assorted sized available

4. REAGENTS AND CHEMICALS

4.1. Organic Solvents

  4.1.1. Methanol - HPLC Grade: CH_3OH - purchased in 4 L amber bottles from Fisher Scientific.
  4.1.2. Methylene Chloride – HPLC Grade: CH_2Cl_2- purchased in 4 L amber bottles from Fisher Scientific.
  4.1.3. Acetone – HPLC Grade: CH_3COCH3 - purchased in 4 L amber bottles from Fisher Scientific.

4.2. Acids

  4.2.1. Hydrochloric Acid, HCl - Fisher Certified ACS Plus Grade.
  4.2.2. Nitric Acid, HNO_3 - Fisher Trace Metal Grade.

4.3. Detergents

  4.3.1. Alconox Powder Detergent
  4.3.2. No-Chrom Cleaning Agent
  4.3.3. LiquiNox Liquid Detergent

4.4. Laboratory DI Reagent Water – Type 1 and Type 2

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5. GENERAL GLASSWARE CLEANING

5.1. Glassware must be scrupulously cleaned. Clean all the glassware as soon as possible after use!

5.2. Rinse with the last solvent used to remove any remaining organic residues. Rinse with hot tap water, then let soak in hot soapy water for one hour.

5.3. Scrub all the sides of each glass container with an appropriately sized brush, rinse with hot tap water, rinse again with deionized water, and give a final rinse with methanol or acetone. Air-dry for 24 hours.

5.4. Store glassware in an inverted position or cover with aluminum foil to insure dust does not enter glassware. Place securely in storage cabinets and drawers.

6. HIGH TEMPERATURE MUFFLE FURNACE CLEANING

All glassware placed in the oven must first be cleaned using the General Glassware Cleaning procedures. Only qualified, trained staff are to use the muffle furnace.

6.1. Glassware must be scrupulously cleaned. Clean all used glassware as soon as possible according to the General Glassware Cleaning procedures in **Section 5**.

6.2. Place the glassware in an area designated for oven-ready glassware and allow to completely air dry

6.3. When dry, place glassware in oven baskets and place the oven-ready glassware baskets on one of the big plastic carts.

6.4. Check the status of the Furnace oven. If baked glassware is already in the oven, then remove the hot glassware from the oven and place it on a metal cart. Push the metal cart with the baked glassware into the Organic Preparation lab. If the glassware is hot, post a “Hot Glassware Caution” sign on the lab door.

Do not allow the baked, hot glassware baskets to come into contact with unbaked aluminum foil. Unbaked aluminum foil has a thin film of organic grease, a PAH contamination source.

6.5. Place the oven-ready glassware into the cool oven. Be careful to place the baskets in the oven gently to avoid damaging the soft oven brick surface. Do not drag the baskets across the brick surface because this will damage the brick.

6.6. The oven must bake the glassware for a minimum of 8 hours above 500°C.

6.7. Make sure the oven maximum operating temperature is set to 550 °C.

6.8. Start the oven controller and exhaust fan with timer.

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6.9. Once the glassware has baked above 500 °C for at least 8 hours, turn off the oven and open the muffle furnace bell with about a 12 inch gap to aid in cooling. Do not open the bell too much since this may stress the glass or present a potential burn risk. Allow the oven to cool for at least 15 minutes and then open the furnace bell fully for at least another 15 minutes. Be careful not to come into contact the oven bell.

6.10. When the oven and glassware have cooled sufficiently for safe handling, unload the oven baskets to metal carts. Push the metal carts to the Organic Preparation Lab for the glassware to cool.. Post a “Hot Glassware Caution” sign on the lab door. Again, do not allow the baked, hot glassware baskets to come into contact with unbaked aluminum foil. Unbaked aluminum foil has a thin film of organic grease, a PAH contamination source.

6.11. Store glassware in an inverted position or cover with baked aluminum foil to insure dust does not enter glassware. Place in storage cabinets and drawers

NOTE: DO NOT PUT VOLUMETRIC GLASSWARE OR GRADUATED CYLINDERS IN THE OVEN

7. CENTRIFUGE TUBES AND OTHER HIGHLY CONTAMINATED GLASSWARE

7.1. Soak all centrifuge tubes in big beakers filled with distilled mixed solvent and sonicate for 1hour.

7.2. Rinse thoroughly with hot tap water, followed by DI water, and then a final rinse with methanol or acetone.

7.3. If the centrifuge tubes or other glassware were used previously for highly contaminated samples, then they must be cleaned separately. Verify cleanliness of the centrifuge glassware by a clean-glassware check.

7.4. Rinse with the last solvent used to remove any residual organic residues. If needed, soak glassware in NO-CHROME Acid Cleaning Solution. Rinse with tap water and then let soak in hot soapy water for one hour.

7.5. Scrub all the sides of glass containers with a proper brush, rinse with hot tap water, rinse again with deionized water and a final rinse with methanol or acetone. Air-dry for 24 hours.

7.6. The glassware's suitability for further use must be verified by a glassware check.

7.6.1. Separate glassware into groups of 10 to 15 pieces per batch.

7.6.2. Rinse each piece of glassware three times with the following solvents and discard in the appropriate waste container: Methanol, Acetone, Methylene Chloride and Hexane.

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7.6.3. Rinse each piece of glassware with 10-20 mL of hexane and collect into a labeled beaker. Use one beaker per batch.

7.6.4. Concentrate the rinsed solvent to 2 to 5 mL and transfer to a GC auto sampler vial, label the batch number, and analyze for all parameters.

7.6.5. Leave all the glassware undisturbed until analyzed. If the glassware passes the check, restore to the shelves. Store glassware in an inverted position or cover with baked aluminum foil to insure dust does not enter glassware.

7.6.6 If any batch does not pass the check, repeat steps 7.2 through 7.5 until the check passes.

8. PIPET CLEANING PROCEDURES

8.1. After using the pipettes, soak them in a pipette container with soapy water. The pipette tips should face upwards.

8.2. Once a week, or when enough pipettes are accumulated, transfer all the pipettes into a pipette rinse with the tips facing upwards.

8.3. Use fresh soapy water for the next collection of dirty pipettes.

8.4. Add 1 or 2 Alconox tablets to the pipette rinse and fill with hot tap water.

8.5. After one hour, wash with hot tap water 4 times and then with deionized water 3 times. Take the wet pipettes and rinse with acetone 3 times and air dry.

8.6. Organize all the pipettes by size and store them in racks for future use. The tips should face downwards.

9. SAMPLING BOTTLE QUALITY CONTROL CHECK

9.1. For every batch of 50 1-liter amber bottles cleaned by the Support Group, one bottle is logged for QC CHECK by assigning W-BNA, W-PSCL-PCB, and W-PEST-NP tests. When four such QC bottles are in the backlog, then test these bottles (glassware check) for contamination.

9.2. Add 10 mL of methylene chloride to each bottle and shake for 3 minutes.

9.3. Decant into a centrifuge tube and split into two equal portions in two centrifuge tubes.

9.4. One portion is solvent exchanged into 5 mL of iso-octane and submit for GC-ECD and NPD analysis for the Laboratory Information Management System (LIMS) tests ID W-PSCL-PCB and W-PEST-NP.

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9.5. Nitrogen blowdown the second portion to 0.40 mL and add 50 µL of BNA internal standard. Make up to 0.50 mL in methylene chloride and submit for GC/MS analysis for LIMS test ID W-BNA.

9.6. Fill out the extraction logbook for either GC or BNA to maintain proper records and change W to V in the LIMS.

9.7. If more than one 1-liter bottle is being checked, then up to 5 bottles may be combined into one sample as a composite.

9.8. For composite samples (up to 5 bottles), add 20 mL of methylene chloride in first the bottle as in step 9.2, and so forth in the subsequent bottles..

9.9. Split the composite extract as done in steps 9.4 and 9.5. Submit the composite sample as one sample, using the first bottle ID for the sample number.

9.10. Enter a comment in the preparation log “Composite with sample numbers….”.

9.11. Cancel the unnecessary samples in the LIMS and enter the same comment as in step 9.10 in the Cancel Comment Field.

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Appendix A Summary of Version Edits

Appendix of changes within this version of the procedure.

- Added Title Page.
- Added Scope and Application section
- Added Method Summary section
- Added Equipment section
- Added Reagents section
- Numbered all sections
- Typo edits to all sections
- Added Appendix Section

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Appendix B Muffle Furnace Programming Instructions

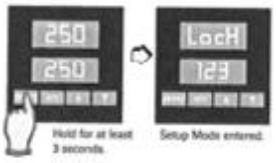
Navigating the furnace's electronic programming requires an understanding of the how the Chromalox 2104 and 3101 controllers work and the definitions of the various program codes. Do not attempt to edit the programming until you have reviewed the procedures and manuals carefully. Refer to the tables below for routine settings.

When the unit is first turned on, the normal Display Mode is shown with current conditions. The controller conditions can be stepped-through by pressing the AUX key.



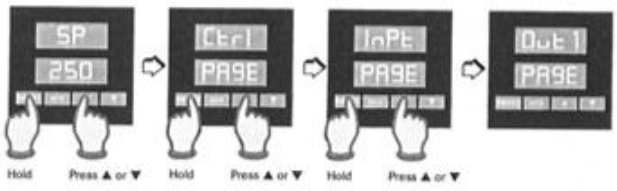
Enter and Exit Setup Mode:

To enter and exit the program set-up mode, press and hold down the RESET button for 3 seconds. The first page displayed is the Control page (displaying the security code). Once in the Setup mode, holding down the RESET key for 3 seconds will exit you from the program, no matter where you are in the program.



Select a Page:

Press and hold the RESET button while pressing the up or down key to increment through the pages available. Release the RESET key when at the desired page. Detailed setting for each of the pages is provided in tables below.

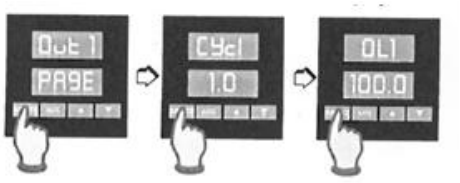


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Appendix B Muffle Furnace Programming Instructions (cont.)

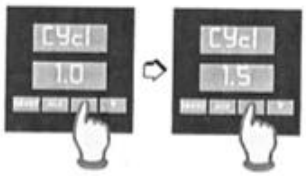
Select a Menu Item within a Page:

Press the RESET Button to scroll through the various menu items.



Change a Menu Value:

After the desired menu is selected and displayed, use the up and down arrow keys to change the value.



Chromalox 2104 Controller Menu Settings:

The tables below contain the normal settings for each page and the menu items contained within each page. Refer to the manual for a detailed description of each menu code.

Control Page – "Ctrl"

Settings on the control page should not normally need modification.

Menu Item	Setting	Menu Item	Setting
LocH	458	FL	On
SP	1	Prn9	0
AuSP	0	Loop	OFF
tune	OFF	Auto	10
Pb1	OnOF	rrAt	OFF
Ar1	.10	Cont	HEAT
rAt1	2	Cool	Pid2
Db1	1	rSP	OFF
Pb2	25	Ent1	nonE
Ar2	.10	Au	nonE
rAt2	10	AOut	ASP
db2	5	rSEn	On
OFSt	0		

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Appendix B Muffle Furnace Programming Instructions (cont.)

Ramp/Soak Page – "rSp9"

The ramp/soak page contains the main ramp and soak program.

Menu Item	Setting	Menu Item	Setting
Unit	hr	int5 through int16	.00
Stby	120	SP5 through SP16	0
int1	.02	cont	oFF
SP1	550	Fro	1
int2	.01	to	1
SP2	550	no	0
int3	12.00	SbEt	nonE
SP3	550	.1E to .16E	nonE
int4	.01	95db	10
SP4	550		

Input Page – "inPt"

Menu Item	Setting	Menu Item	Setting
SenS	H	SPLL	0
Unit	°C	SPUL	800
CoFF	0		

Custom Input/Output Scaling Page – "ScAl"

Menu Item	Setting	Menu Item	Setting
dP	1	AotH	1316
AinL	.0	rSPL	-184
AinH	100.0	rSPH	1316
AotL	-184		

Output #1 Page – "Outl"

Menu Item	Setting	Menu Item	Setting
CYcl	5.0	HoFF	0
OL1	100.0		

Output#5 Page – "Out5"

Menu Item	Setting	Menu Item	Setting
tYP5	OFF	AHi5	1316
ALr5	nonE	db5	1
rLY5	ndE	Inh5	OFF
ALo5	-184		

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Appendix B Muffle Furnace Programming Instructions (cont.)

Digital Communication Page – "di9"

Menu Item	**Setting**	**Menu Item**	**Setting**
Digt	CPiF	Addr	1
bAud	19.2H		

Chromalox 3101 High/Low Limit Controller Menu Settings:

The tables below contain the normal settings for each page and the menu items contained within each page. Refer to the manual for a detailed description of each menu code.

Limit Control Setup Page – "SEt"

Menu Item	**Setting**	**Menu Item**	**Setting**
LocH	458	db	1
L SP	570		

Input Page – "inPt"

Menu Item	**Setting**	**Menu Item**	**Setting**
SenS	H	SPLL	10
Unit	°C	SPUL	570
CoFF	0		

Alarms #1 Page – "AL1"

Menu Item	**Setting**	**Menu Item**	**Setting**
En1	On	AHi1	580
tYP1	Hi	db1	100
rLY1	ndE	Inh1	On
ALo1	-184		

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Appendix C: Posted Operating Instructions at Instrument

Wilt 210 Muffle Furnace Operating Instructions

1) The oven temperature is viewable as the top red-colored readout on the 2104 Controller (left side controller).



2) Turn the oven switch to the “OFF” position before opening the oven.

3) The oven cover may be lifted 6-12 inches to facilitate faster cooling.

4) Wait until the oven has comfortably cooled before removing and load glassware. Metal or glassware items must not contact the oven wall.

5) Turn the oven switch to the “ON” position.

6) Resetting the 2104 Controller: press both the “UP” and “DOWN” arrow keys simultaneously to abort the old program. The Green readout should read the standby temperature at 120°C.

7) Starting the 2104 Controller Program: press the “UP” arrow once to restart the oven program. The green readout should quickly increase to 550 °C and stay at 550 °C.

8) By pressing the “AUX” key three times you may view the time remaining, which should read 12.00 hours.

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