# **Table of Contents**

1.	Pur	pose			
2.	2. General Information				
3.	Pro	ocedure			
3	8.1.	Agilent Instruments			
3	3.2.	Thermo Scientific	6		
4.	Dis	screpancies			
5.	Ref	ferences	9		

SOP Name:		SOP #:
GC MS Maintenance		061
	Revision:	Revision Date/Initials:
North Carolina Office of the Chief	3.1.1.1 – Updated with NPD instructions 3.1.12 - Updated with NPD instructions	MSF 05/03/2016
Medical Examiner Toxicology Laboratory		
Approving Authority Name	Approving Authority Signature	Approval Date
Ruth E. Winecker, Ph.D.	Stattellinder	04/15/2015
Ruth E. Winecker, Ph.D.	Stuttellinder	06/27/2016
Ruth E. Winecker, Ph.D.	Puttellinden	12/06/2017
$c \nabla$		
S		

#### 1. Purpose

1.1. This procedure is designed to establish the minimum requirements to perform weekly inlet maintenance for Agilent and Thermo Scientific GC/MS, GC/NPD, GC/FID analytical equipment.

NOTE: The operating temperatures of the instruments discussed below are > 200°C. A burn hazard exists when handling injection and detector assemblies. Use great care.

#### 2. General Information

2.1. Schedule



- 2.1.1. Unless otherwise noted, routine inlet maintenance should be performed on instruments performing a normal workload every week (usually in Monday).
  - 2.1.1.1. If the instrument was not used the previous week then the log is documented: "instrument not in use" and maintenance is not performed.
  - 2.1.1.2. If an instrument is not used for greater than 2 weeks then maintenance will be performed prior to next batch to be run.
  - 2.1.1.3. Maintenance may need to be performed more often than once per week if heavily used or damaged by dirty extracts.

#### 3. Procedure

- 3.1. Agilent Instruments
  - 3.1.1. For parts and diagrams refer to: <u>Agilent 6890 Manual</u>
    - 3.1.1.1. Using the GC keypad set the inlet temperature to 'Off' and oven temperature to 50°C. Wait until the inlet temperature falls below 100°C before beginning maintenance. In addition, for instruments utilizing NPD detectors, do the following:
      - 3.1.1.1.1. Turn detector temperature to 90 °C
      - 3.1.1.1.2. Set bead voltage to 1.0 V
      - 3.1.1.1.3. Turn electrometer off.
  - 3.1.2. Replace Septum
    - 3.1.2.1. Unscrew the inlet cap (with fingers or small end of Agilent inlet tool).
    - 3.1.2.2. Using tweezers or other sharp implement, pry the used septum out of its housing and press a new septum in its place.
    - 3.1.2.3. Screw inlet cap back on (finger tight).
  - 3.1.3. Remove Inlet Liner

- 3.1.3.1. From the keypad of the GC, turn the inlet flow off.
- 3.1.3.2. Using the large end of the Agilent inlet tool unscrew the weldment from the inlet (counterclockwise).
- 3.1.3.3. Remove old liner and O-ring.
- 3.1.3.4. Use a cotton swab saturated with methanol to clean the inlet around the O-ring and bottom of the weldment. Also clean the threads of the weldment and top of the inlet housing.
- 3.1.4. Replace Gold Seal and washer:

NOTE: GCMS instruments with capillary columns require gold seal changes weekly but on GCFID or GCNPD instruments with megabore columns, gold seals should only be changed monthly in heavily used inlets and on an as needed basis for infrequently used inlets.

- 3.1.4.1. Open the oven.
- 3.1.4.2. Using a <sup>1</sup>/<sub>4</sub> in. crescent wrench, unscrew the inlet nut (clockwise) and remove the column and column nut from the inlet housing.
- 3.1.4.3. Unscrew the side screws then twist to release the insulation cup.
- 3.1.4.4. Using a <sup>1</sup>/<sub>2</sub> in. crescent wrench, unscrew the exposed reducing nut (clockwise).
- 3.1.4.5. Remove the old gold seal and washer. Clean out the thread of the reducing nut with a cotton swab saturated with methanol.
- 3.1.4.6. Insert a new washer, and then a new gold seal with the nipple facing down.
- 3.1.4.7. Screw the reducing nut back into place, install the insulation and tighten the insulation cup screws.
- 3.1.5. Replace inlet liner and O-ring.
  - 3.1.5.1. Place a new O-ring onto a new inlet liner and slide to the mid-point.
  - 3.1.5.2. Insert liner into inlet, open-side up, and press firmly into place.
  - 3.1.5.3. Replace the weldment on the inlet, turning the weldment nut clockwise until firmly tightened.

- 3.1.6. Trim and reinsert column
  - 3.1.6.1. Score and break off 5 to 33cm of the inlet end of the column (depending on use).
  - 3.1.6.2. Slide column nut onto the column threads facing toward the inlet end.
  - 3.1.6.3. Slide a new ferrule onto the column, flat side facing the column nut.
  - 3.1.6.4. Score and break off 2cm of column. Visually inspect the end of the column to ensure a clean break, repeat if necessary.
  - 3.1.6.5. Leave 5 mm of the column exposed to the inlet from the inlet nut.
  - 3.1.6.6. Screw the inlet nut with the column back into the inlet  $-\frac{1}{2}$  to  $\frac{3}{4}$  turn past finger tight.
- 3.1.7. Record all maintenance performed in the log book for that instrument.
- 3.1.8. Turn the inlet flow on.
- 3.1.9. Turn the inlet temperature to 'On'
- 3.1.10. Set oven temp to 300°C
- 3.1.11. Wait 15 minutes.
- 3.1.12. For NPD instruments:
  - 3.1.12.1. Make sure that the air and hydrogen gases are set to "ON".
  - 3.1.12.2. Turn the bead temperature to 150° C.
  - 3.1.12.3. Wait 15 minutes.
  - 3.1.12.4. Turn the bead temperature to 200° C.
  - 3.1.12.5. Wait 15 min.
  - 3.1.12.6. Turn the bead temperature to 250° C.
  - 3.1.12.7. Wait 30 minutes.
  - 3.1.12.8. Turn the bead temperature to operating temperature (290 °C).
  - 3.1.12.9. When the bead temperature has stabilized turn the electrometer to the

"ON" position. Check that the output is below 1.2pA by pressing the detector button on the gc and scrolling down to view the output.

- 3.1.12.10. If the output is above 1.2pA cool the bead and replace the ceramics around the bead. Reinstall the bead and screws and repeat steps 2.9 thru 2.17.
- 3.1.12.11. Turn the bead voltage to 1.00V. Hold for 2 minutes.
- 3.1.12.12. Turn the bead voltage to 1.5V. Hold for 2 minutes.
- 3.1.12.13. Turn the bead voltage to 2.0V. Hold for 2 minutes.
- 3.1.12.14. Turn the bead voltage to 2.2V. Hold for 2 minutes
- 3.1.12.15. Turn the bead voltage to 2.4V. Hold for 2 minutes
- 3.1.12.16. Turn the bead voltage to 2.6V. Hold for 2 minutes
- 3.1.12.17. Turn the bead voltage to 2.8V. Hold for 2 minutes
- 3.1.12.18. Watch for the bead output to "ignite" (bead output jumps quickly to a higher pA typically over 50pA)
- 3.1.12.19. When the bead "ignites" do not change the bead voltage and let it equilibrate for 12 hours.
- 3.1.12.20. After 12 hours inject a testmix and evaluate the baseline, response, separation, etc. for acceptability.
- 3.1.13. Load the instrument method on Chemstation software (online instrument).
- 3.1.14. Wait until all temperatures and pressures reach equilibrium before running the autotune

3.1.15. Run Auto-Tune

3.1.15.1. See SOP 060 for instructions on performing the tune and evaluation.

#### 3.2. Thermo Scientific

- 3.2.1. Routine inlet maintenance should be performed as needed or, at a minimum, every 7 working days.
- 3.2.2. Turn the inlet temperature off and the oven temperature to 50 °C. Wait until the inlet temperature falls below 100°C prior to starting maintenance.

- 3.2.3. Replace Septum.
  - 3.2.3.1. Turn inlet pressure off.
  - 3.2.3.2. Unscrew the septum cap.
  - 3.2.3.3. Pull off the septum holder.
  - 3.2.3.4. Replace the old septum with the new septum.
- 3.2.4. Remove Liner
  - 3.2.4.1. Use the liner cap removal tool to remove the liner cap.
  - 3.2.4.2. Use the Thermo tweezers to remove the glass liner and graphite liner seal.
  - 3.2.4.3. Remove the graphite liner seal and place it on a new liner. Set aside.
- 3.2.5. Silver liner
  - 3.2.5.1. Unscrew the fixing nut for the column at the inlet end of the GC column.
  - 3.2.5.2. Remove the column and plug the end with a used septum.
  - 3.2.5.3. Unscrew the terminal nut fitting.
  - 3.2.5.4. Remove the terminal fitting.
  - 3.2.5.5. Replace the old silver seal that the base of the terminal fitting with a new silver seal.
  - 3.2.5.6. Replace the terminal fitting.
  - 3.2.5.7. Replace the terminal nut fitting.

#### 3.2.6. Install New Liner

- 3.2.6.1. Insert the new liner with graphite seal into the inlet.
- 3.2.6.2. Insert the liner cap and tighten down with the liner cap removal tool.
- 3.2.6.3. Replace septum and septum holder.
- 3.2.6.4. Tighten down the septum cap very lightly until it resists tightening

slightly.

- 3.2.7. Trim column
  - 3.2.7.1. Unscrew fixing nut from the bottom of the inlet exposing the column end inserted into the inlet.
  - 3.2.7.2. One the inlet end of the column, score and break off 5-33 cm of column (depending on use/chromatography).
  - 3.2.7.3. Replace fixing nut if needed.
  - 3.2.7.4. Use Thermo three-sided ruler to adjust the column to the appropriate height and reinsert into the inlet
  - 3.2.7.5. Tighten the fixing nut with column back into the inlet.
  - 3.2.7.6. Record all maintenance performed in the log book for that instrument.
  - 3.2.7.7. Turn the inlet flow back on.
  - 3.2.7.8. Turn Inlet temperature to 'On
  - 3.2.7.9. Set oven temperature to 300°C.
  - 3.2.7.10. Wait 15 minutes.
  - 3.2.7.11. Load the instrument method from the computer to the instrument.
  - 3.2.7.12. Wait until all temperatures and pressures reach equilibrium before running the autotune.
- 3.2.8. Run Autotune:
- 3.2.9. See SOP 060 for instructions on performing the tune and evaluation

### 4. Discrepancies

**4.1.1.** If there are any problems during inlet maintenance or in the assessment of the tune or testmix after performing inlet maintenance, bring the data to the attention of the instrument or other senior chemist.

## 5. References:

- 5.1. Agilent 6890 GC System Installation Guide
- 5.2. Agilent 6890 GC System Users Guide
- 5.3. Thermo DSQ I/II System Users Guide
- 5.4. Thermo GC Troubleshooting Guide