

**FORENSIC TOXICOLOGY LABORATORY
OFFICE OF CHIEF MEDICAL EXAMINER
CITY OF NEW YORK**

GC/MS MAINTENANCE

TANK REPLACEMENT

A GC/MS helium carrier gas tank requires replacement when the pressure of the tank is 200 psi or less.

Materials

Weak soap solution in a squeeze bottle (e.g., "Snoop")

Open-ended adjustable wrench which is able to open to at least a 2 inch diameter

Procedure

1. On GC control panel reduce heated zones to 50 °C. (Injector, detector, and oven). Wait for temperatures to fall to the set point.
2. On the gas tank, turn the flow control knob in the "close" direction until the tank is fully closed.
3. Using the open-end adjustable wrench, loosen the large nut connecting the gas regulator to the tank outflow. Remove the gas regulator.
4. Place a steel protective cap onto the empty gas tank. Label as empty. Remove the tank restraints and chain the empty tank in the gas storage area.
5. Move a new, full tank into place and secure it with the tank restraints. Place the gas regulator onto the tank outflow and tighten the nut using the wrench. Do not over-tighten. Rotate the flow control knob on the tank outlet in the "open" direction until tank is fully open.
6. Dispense the weak soap solution onto all joints and connections. The formation of soap bubbles indicates a gas leak. Tighten or replace swage joints and connections as necessary.
7. On the GC control panel, return heated zones to their proper set points.
8. Return all tools to their proper location.

SEPTA REPLACEMENT

The septum in the GC requires replacement when there is a significant change in the retention time of known peaks in any given control or an increase in baseline signifying a “leak” in the system. As a practical matter, it is recommended that a septum be changed on a weekly basis or after approximately 200 injections.

Materials

One pair of thermally resistant gloves

One septum nut wrench suitable for 15mm hexagonal nuts

Tweezers

Cotton swabs

Heptane

Replacement septum (11mm diameter and 3mm thick)

Procedure

1. Remove injector tower from top of GC.
2. Locate the 15mm hexagonal nut underneath the previously mounted position of the tower. Using the septum nut wrench, turn the septum retainer nut in a counter clockwise direction until the nut spins freely. (Due to thermal expansion, the nut may be difficult to move at first.)
3. Remove the retainer nut

NOTE: MAY BE EXTREMELY HOT!

4. Using the tweezers, remove the old septum from the inlet. Discard.
5. Soak a cotton swab in heptane. Use this to clean the inlet of build-up or debris. Using tweezers, remove any particulate matter from the inlet area.
6. Locate a new septum and, using the tweezers, place the new septum firmly in the inlet. Replace the septum retainer nut and turn in a clockwise direction using the 15mm hexagonal septum nut wrench. Be certain not to over-tighten.
7. Replace the injector tower. Record this maintenance in the maintenance log.

CARBO-FRIT AND INJECTION PORT LINER REPLACEMENT

The carbo-frit and liner in the GC requires replacement when the chromatogram of a control sample exhibits the failure of appearance of any given drug or relative abundances are lower

than normal, or drug peaks exhibit irregular symmetry. On the average, liners should be replaced weekly for proper chromatography.

Materials

One pair of thermally resistant gloves

Insert retainer nut wrench suitable for 22mm hexagonal nuts (insert retainer nut)

Cotton swabs

Tweezers

Clean glass splitless liner

Carbo-frit

Puller/packing tool

Procedure

1. Remove injector tower from top of GC.
2. Locate the 22mm insert retainer nut. This is the larger nut that resides beneath the septum retainer nut. Using the insert retainer nut wrench, loosen the insert retainer nut by rotating it in a counter-clockwise direction.
3. Once the nut is free of the threading, *gently* move it aside. Be extremely careful not to snap or bend the tubing connected to the nut.
4. Using tweezers lift the liner straight up until it is free of the injection port. Do not bend or twist the liner during removal as doing so may cause the liner tube to crack or chip in the injection port.

NOTE: CAUTION! LINER MAY BE EXTREMELY HOT!

5. Once the glass liner is free of the injection port, place it on a clean, flat surface. A graphite o-ring will be at the head of the liner. Simply remove this and place it on a clean, flat surface.
6. Using the puller/packer tool, push the carbo-frit out of the glass liner tube. Discard the old frit. Place the used glass liner into a container marked "used glass liners."
7. Acquire a clean, unused glass liner. Obtain a new carbo-frit and place it in the mouth of the new liner. Using the packer/puller tool, push the carbo-frit into the glass liner. Pack the plug so that it is approximately 1cm from the end of the glass liner that will be against the gold seal in the injection port.
8. Rinse liner through with methylene chloride (from each end of liner).

9. Place the non-stick fluorocarbon o-ring on the end of the glass liner. Insert the liner back into the injection port such that the frit end enters the injection port first.
10. Replace the insert retainer nut and tighten it in a clockwise direction using the insert retainer nut wrench. Replace the tower.
11. Immediately record this maintenance in maintenance log.

GLASS LINER CLEANING AND PREPARATION

Once removed from the injection port of the GC, used glass liners should be placed into a properly labeled container for cleaning. When required for maintenance, these used liners should be properly cleaned and silanized for reuse in the GC.

One pair of lint free gloves

Tweezers

Squeeze bottle with deionized water

Squeeze bottle with methanol

Squeeze bottle with ethanol (100%)

Separate containers (each large enough to hold several liners) filled with concentrated nitric acid

Heptane

Dichlorodimethylsiloxane (5% solution by volume with toluene)

Toluene

Procedure

1. Using the tweezers, place all used liners into a container filled with concentrated nitric acid. Cap this container and allow the tubes to soak in the acid for a minimum of one hour.
2. Remove each tube from the acid bath one at a time (using the tweezers) and rinse each tube with the deionized water from the squeeze bottle. Follow this with a methanolic rinse and place the tube into a container partially filled with heptane. Continue this until all tubes are in the heptane container. Make certain that there is enough heptane to completely cover all of the tubes.
3. Allow the glass liners to soak in the heptane for ten minutes. During this time, gently invert the container once every two to three minutes.
4. Using the tweezers, remove all liners from the heptane container and place all tubes into a container partially filled with dichlorodimethylsiloxane (5%). Make certain there is enough

dichlorodimethyl-siloxane to completely cover all of the tubes. Allow the tubes to soak for a minimum of thirty minutes. During this time, gently invert the container once every two to three minutes.

5. Remove each tube from the dichlorodimethylsiloxane bath one at a time (using the tweezers) and place each tube into a container filled with toluene. Make certain that there is enough toluene to completely cover all of the tubes. Cap the container and allow the tubes to soak in the toluene for ten minutes. During this time, gently invert the container once every two to three minutes.
6. Using the tweezers, remove each tube from the toluene bath one at a time and rinse each tube with the 100% ethanol. Place the tube into a container partially filled with heptane. Repeat this procedure until all tubes are in the heptane container. Make certain that there is enough heptane to completely cover all of the tubes. Cap the container and allow the tubes to soak in the heptane for ten minutes. Gently invert container once every two to three minutes.
7. Using the tweezers, remove the liners from the heptane bath one at a time and allow liners to briefly air dry. Store the liners in a desiccator until required for maintenance of GCs.

GOLD SEAL AND WASHER REPLACEMENT AND CLEANING

Once removed from the injection port of the GC, used seals and washers should be placed into a properly labeled container to await cleaning. When required for maintenance, these used seals should be properly cleaned and sonicated for reuse in the GC.

1. Gold seals and washers should be cleaned with Wenol[®], if available, or aluminum silicate combined with methanol to form a paste, on a cotton tipped applicator to remove deposits.
2. The seals and liners should then be placed in a beaker containing methanol and sonicated for 5 minutes.
3. The methanol should be drained to waste and then 1,1,1-trichloroethanol should be added and seals sonicated for 5 minutes.
4. Drain the 1,1,1-trichloroethanol to waste and add methanol for a second time and sonicate for 5 minutes.
5. Drain the methanol and dry seals and washers in a 100 °C oven for 15 minutes. Seals and washers are ready for use.

SOURCE REMOVAL AND CLEANING

See appropriate maintenance manuals supplied by manufacturer. The Agilent MSD reference collection CD has a video clip showing proper source cleaning procedures.

MAINTENANCE SCHEDULE FOR GC/MS LAB

1. Septa - Replace approximately every 200 injections

2. Injection port liner - Replace weekly unless poor chromatography warrants changing sooner.
3. Gold injector seal and washer - Change monthly with injection port liner or as needed.
4. Gas Tanks - Change when pressure in tank has dropped to around 200 psi.
5. Columns - Change when poor chromatography warrants the need.
6. PFTBA calibrator - Change when abundances on autotune seem low or reservoir seems to be almost empty.
7. Source cleaning - Clean source when system fails autotune or peak symmetry in manual tune becomes poor.

REFERENCES

Agilent 6890 GC System Installation Guide.

Agilent 6890 GC System Users Guide.

Agilent 6890 GC System Standard Operating Procedures.

Agilent 5973 & 5973 Network Mass Selective Detector Installation Guide.

Agilent 5973 & 5973 Network Mass Selective Detector Users Guide.

Agilent 5973 & 5973 Network Mass Selective Detector Standard Operating Procedures.

Agilent 5973 MSD Reference Collection CD.