

MAINTENANCE AND CLEANING OF THE GCS.
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As the stationary phase is degraded by the interaction with reactive analytes, the resolution of the capillary columns will gradually degrade. It is possible to partially restore the resolution by reconditioning the columns with either a commercial conditioning agent, or a home made conditioning solution.

If noticeable tailing occurs with a standard test solution of normal hydrocarbons, the column needs to be reconditioned. Tailing of polar compounds is normal for the nonpolar phase used on our columns, though. Since both operations require cooling the instrument down, it makes sense to clean and repack the injector liner at the same time. The reconditioning and cleaning procedure is as follows:

1. Set Inj A temperature to OFF.
2. Push Sig A twice and set it to OFF.
3. Set Det A temperature to OFF.
4. Set Oven Temp to OFF.
5. Let the instrument to cool down overnight.
6. Remove the septum retainer nut.
7. Remove the old septum. It may be stuck to the base and require careful scraping with a screwdriver.
8. Loosen the large nut holding the lid of the injection port and remove the lid. It will remain hanging around by two stainless steel gas lines.
9. Carefully pry the old O-ring loose from the metal base with a small screwdriver.
10. Carefully remove the brittle glass liner from the injection port.
11. Reinstall the injection port lid without the liner.
12. Install a new septum.
13. Reinstall the septum retainer nut.
14. Remove and discard the old O-ring.
15. Remove the old packing with a hooked piece of thin wire. An average size straightened paper clip will do fine. Note the amounts of glass wool and silica used.
16. Inspect the inner surface of the insert for contamination. Clean with acetone or piranha solution, if necessary.
17. If the insert was wet cleaned, rinse it carefully and dry in the oven overnight before repacking.
18. Repack the insert with the same amounts of silanized glass wool and silanized silica (or silanized Chromosorb) as were removed in # 15. The glass wool should be packed carefully, but tightly.
19. Inspect the inside surface of the FID for contamination (soot) by looking through the exhaust port and using a flashlight, if necessary.

If the FID appears contaminated, or if the baseline signal level is unacceptably high, even though no residue can be seen, remove, disassemble and clean the detector (see below). Put a piece of aluminum foil over the detector opening in the housing of the GC to prevent dust accumulation. Bake the parts of the detector (EXCEPT THE PLASTIC INSULATED WIRE) in the oven at 150-200°C overnight. Reassemble and reinstall the detector as follows.

1. Remove the injection port lid as in # 8 above.
2. Put a new O-ring on the repacked liner at ca. 10 mm from its top end.
3. Insert the liner into the injection port.
4. GENTLY push the liner in, until it touches the bottom of the injection chamber.
5. CAREFULLY reinstall the injection port lid. The nut should be tightened with fingers ONLY, or the liner may be crushed. When the nut is finger tight, an additional ¼ of a turn may be applied with a wrench, if the column head pressure doesn't reach the desired level.
6. Set Inj A temperature to 270°C.
7. Set Sig A to ON.
8. Set Det A temperature to 300°C.
9. Set Oven Temp to 250°C.
10. When the red (NOT READY) light is out, push the igniter button.
11. Let the GC bake off overnight.
12. Recondition the column with 3-5 injections of the silanizing solution (10 µL each) keeping the oven at 250°C.
13. Let the GC bake off overnight.
14. Run a test sample of normal hydrocarbons (50°C to 250°C, rate 10°C/min, initial time 2 min).

FID cleaning instructions.

1. Remove 3 Phillips screws holding the detector to the housing of the GC.
2. Disconnect the snap connector holding the insulated wire.
3. Remove the detector.
4. With the Pozidriv wrench (supplied) carefully remove the jet that can be seen deep within the opening remaining after the detector removal.
5. Loosen the nut holding the insulated wire to the housing of the detector and remove the wire and the copper washer. Set them aside.
6. Remove large brass round nut.
7. Disassemble the rest of the detector paying close attention to the way its put together.
8. Prepare 250 mL of a suspension of 2 parts saturated Alconox solution, 2 parts deionized water and 1 part silica gel.
9. Put all metal parts of the detector (EXCEPT THE INSULATED WIRE) and the jet in a 200-mL beaker and cover them with the silica suspension from # 8.
10. Put all Teflon inserts in a SEPARATE 200-mL beaker and cover them with the silica suspension from # 8.
11. Sonicate the parts in an ultrasonic cleaner for 30 min, turning them in the suspension occasionally.
12. Remove the parts from the suspension and rinse them with deionized water 5 times. Make sure the jet is clear of silica.
13. Put the parts (EXCEPT THE INSULATED WIRE) on a sheet of aluminum foil and dry them in the oven at 150-200°C overnight.
14. Clean ONLY the metal tip of the insulated wire with ethanol and allow it to dry at 50-70° overnight.
15. Reassemble the detector wearing talcum free rubber gloves.

16. CAREFULLY reinstall the jet, making sure the tip of the column goes into the stem of the jet before letting it fall down.
17. Gently tighten the jet with the Pozidriv wrench.
18. Reinstall the detector.
19. Reconnect the insulated wire.

Preparation of silanized glass wool.

1. Pack a beaker tight with regular glass wool.
2. Cover the wool with the silanizing solution (see below) and keep it immersed for 30 min.
3. Drain the solution.
4. Wash the wool with ethanol (3X) and deionized water (3X).
5. Dry the wool at 150-200°C overnight wrapped loosely in a piece of aluminum foil.
6. Store the silanized wool in closed jar.

The suggested solution consists of 1 volume of chlorotrimethylsilane, 2 volumes of hexamethyldisilazane and 6 volumes of hexane. The solution is EXTREMELY moisture sensitive and is best prepared immediately prior to use. Moderate cloudiness is acceptable. Active solution should immediately render any clean glass surface hydrophobic.