MSD EI and CI Source Cleaning and Installation

Technical Overview

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Introduction

During the course of operation, eventually GC-MSD sources require cleaning. This can be indicated by a loss in analyte response that is not improved by GC inlet and column servicing; or in source tuning by poor calibrant ion peak shapes or escalating repeller or electron multiplier voltages. Consider Figure 1, which shows the difference in response for several pesticides before and after proper source cleaning.

Proper cleaning, assembly, and installation are essential to robust and reliable operation, especially for the assembly of the CI ion source. A variety of source cleaning methods exist in laboratories around the world, some advocating polishing compounds, soaps, or harsh agents. These most often work for a very limited application range and never are successful over the long-term performance of the source. For example, most polishing compounds and some soaps contain high-molecular-weight waxes, which produce a raised background and lowered response. Chemical agents can adversely affect the source surface chemistry and increase activity.

Based on many years of experience, this overview describes a simple approach to cleaning and reinstalling the 5973 and 5975 ion electron impact (EI) inert and standard sources and the chemical ionization (CI) source.

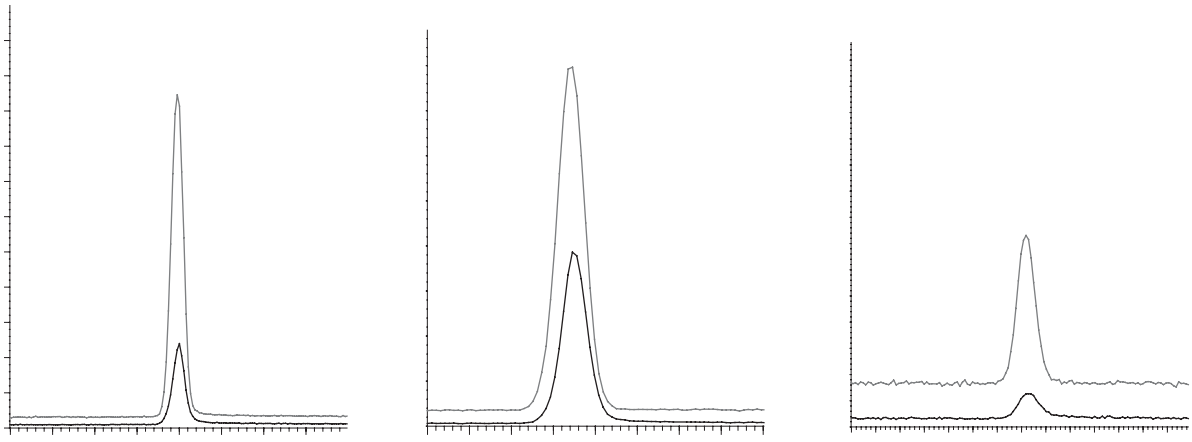


Figure 1. Overlaid RTICs for Azinphos-ethyl (left) and the Demeton isomers (right) before (lower trace) and after (upper trace) mechanical cleaning.

Precautions

Like all laboratory work involving flammable or potentially toxic solvents, the appropriate precautions should be taken.

All solvents, glassware, and foil must be clean and free of contamination and exchanged as necessary during the cleaning process.

Before starting a source cleaning, consumables, such as the filaments, or spare parts, such as repeller ceramics, should be on hand.

Cleaning Procedure

Source disassembly is described in the hardware manual and associated video (refer to Agilent 5973 and 5975 Series MSD Hardware User Information DVD). It is important that all ceramics, the entrance and ion-focus lenses insulator, the source heater block, all screws, and the filaments are always placed on a clean, lintless material (like solvent-rinsed or fired foil) and are never exposed to any solvent (refer to Figure 2). Separating the metal parts for cleaning makes this easier (Figures 2 and 3).

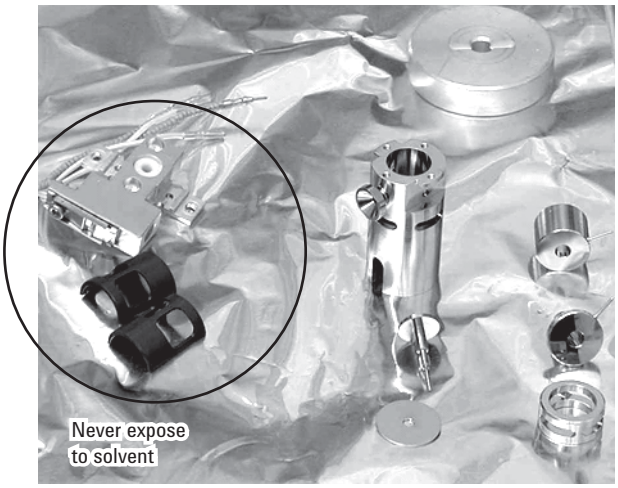


Figure 2. Disassembled and separated EI source components.

In the past, the green paper was recommended for cleaning sources. While this is still applicable to the Agilent 5971 and 5972 MSD Series, it is not the preferred method of cleaning for the newer sources of the 5973 and 5975 MSD Series. Instead, apply the following approach.

1. Make a slurry or paste of the alumina MICROGRIT supplied with the instrument by adding deionized water to a small amount of the MICROGRIT powder (refer to Figure 4). The consistency should be a little thick.
2. Use the supplied cotton-tipped swabs to apply a small amount of the MICROGRIT-water paste to the metal components. Rubbing will remove enough material to produce a clean and shiny surface.

   In cleaning the source bodies of the EI and CI sources, it is important to also clean the filament holes. Use a wooden toothpick to apply the MICROGRIT paste to the holes and also to remove or clear MICROGRIT from the filament holes as the last step in cleaning the source bodies.

   The parts most prone to contamination are the source body, repeller, and drawout lens. These should be cleaned with special attention.
3. Rinse all parts in deionized water. Remove as much MICROGRIT as possible at this stage.
4. Submerge all the rinsed parts in a beaker of deionized water and sonicate for about 5 minutes. Steps 4, 5, 6, and 7 refer to Figure 5.

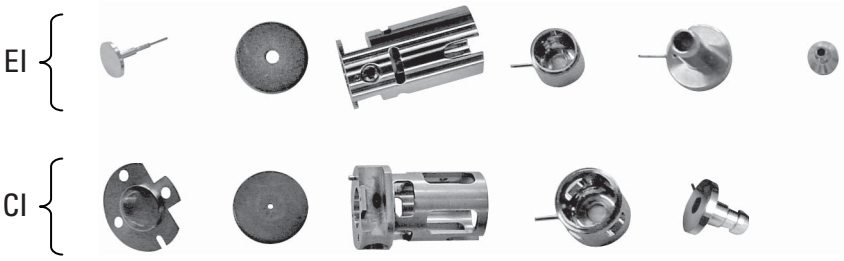


Figure 3. EI and CI Source metal components for cleaning.

5. Use metal tweezers to remove the parts carefully from the beaker of water and submerge them in a beaker of methanol (pesticide or HPLC grade). Sonicate for about 5 minutes.
6. Use metal tweezers to remove the parts carefully from the beaker of methanol and submerge them in a beaker of acetone (pesticide or HPLC grade). Sonicate for about 5 minutes.
7. Use metal tweezers to remove the parts carefully from the beaker of acetone and submerge them in a beaker of hexane (pesticide or HPLC grade). Sonicate for about 5 minutes.
8. Use metal tweezers to remove the parts carefully from the beaker of hexane and place them on clean foil or lint-free tissue. Carefully

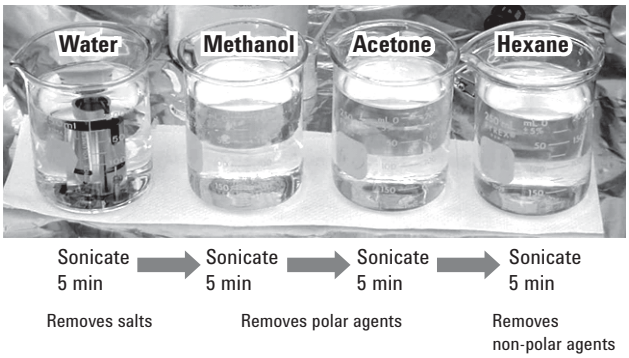
Figure 4. Microgrit and slurry.

reassemble the source as instructed in the manual. Inspect the filaments and replace them if they look worn. Be especially careful with the ceramics for the repeller to ensure that they are not cracked, which usually happens if they have been overtightened.

9. Immediately reinstall the source in the MSD. Do not bother baking the source in an oven as the source should be very dry after the acetone rinse and free of organics after the hexane rinse, which rapidly evaporates. Only residues in your solvents will remain, hence the requirement for very clean solvents.

Reinstallation Procedure

If the GC column is being installed, it should be properly conditioned outside the MSD first and then installed through the MSD transfer line. After extending through the transfer line, it should be trimmed and pulled back until just 1 mm extends past the tip of the transfer-line interface (Figure 6). Tighten the transfer-line ferrule until



Solvents must be very clean. Especially the hexane!

Figure 5. Ion Source cleaning sequence.

two squeaks are heard. Remember that after the transfer line has been heated, the ferrule will have to be tightened again. It should be checked for tightness consistently as this is a major source of leaks when the transfer line is temperature cycled. Another approach is to use the SilTite metal ferrules (part number 5184-3569 for 0.2- to 0.25-mm id columns). These ferrules must be carefully tightened. When properly installed, they form a stable seal that does not require constant readjustment after temperature cycling.

An ion source should never be heated until it is established that there is a "good" vacuum. This means checking that there are no leaks and that the water has been lowered. Heating a source to operating temperatures in the presence of a leak or high-water background can activate it, essentially undoing the effort of the cleaning process. A better procedure is to pump down the system with the source, quad, and GC zone temperatures cold (i.e., set to 0 °C), wait 20 minutes, and examine.

1. Does the vacuum gauge indicate the manifold pressure is coming down? (Or is the foreline pressure dropping toward operating values?)
2. Then, if pressures look good, enter the Manual Tune panel and examine the air and water values (Figure 7). If nitrogen (28 *m/z*) has dropped to < 25,000 counts absolute, then the system is mainly airtight. Water will drop more slowly and requires system baking.
3. Load the analytical method.

The philosophy for operating in this manner is that if there is a leak, the instrument zones are still cold, so the user can troubleshoot immediately without waiting for the zones to cool.

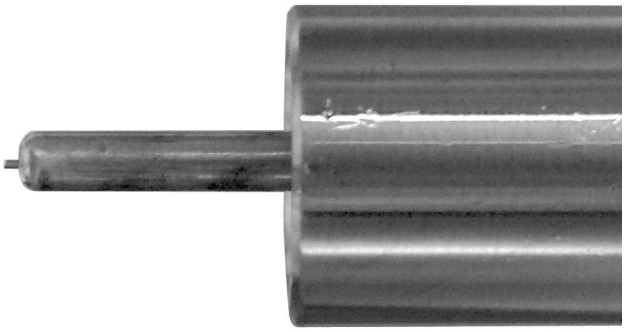


Figure 6. Installed and conditioned column in MSD. Pull back column until 1 mm extends beyond the interface tip.

Analyzer Baking

A useful macro allows “baking” of the source and quadrupole to lower water background and improve performance in tuning and acquisition. This requires that the tune parameters allow higher source temperatures and these should be set to the maximums allowed: 300 °C for the ion source and 200 °C for the quad (in the Manual Tune Panel or from the Source Temperature Panel) and saved (Figure 8).

The macro Bake.mac is called as macro "bake.mac",go <enter> from the command line. The user is prompted for the baking temperatures and the duration of the bake (refer to Figure 8).

Bake.mac should be loaded or located under the \msdchem\msexe directory. This macro requires MSD ChemStation G1701DA rev D03 or higher.

If the macro is not located under the directory users may obtain it from the Agilent Web site if they login as users and have a registration number for their software. More detailed instructions are given below.

Automated Baking and Performance Checkout

An automated approach uses the macro Bake.mac. Here the user has pumped the system down, checked that the system is leak-free, and loaded their method and this checkout sequence. Typically this Checkout.M method is used to confirm the performance of the instrument after a servicing. This macro operates in a sequence such as

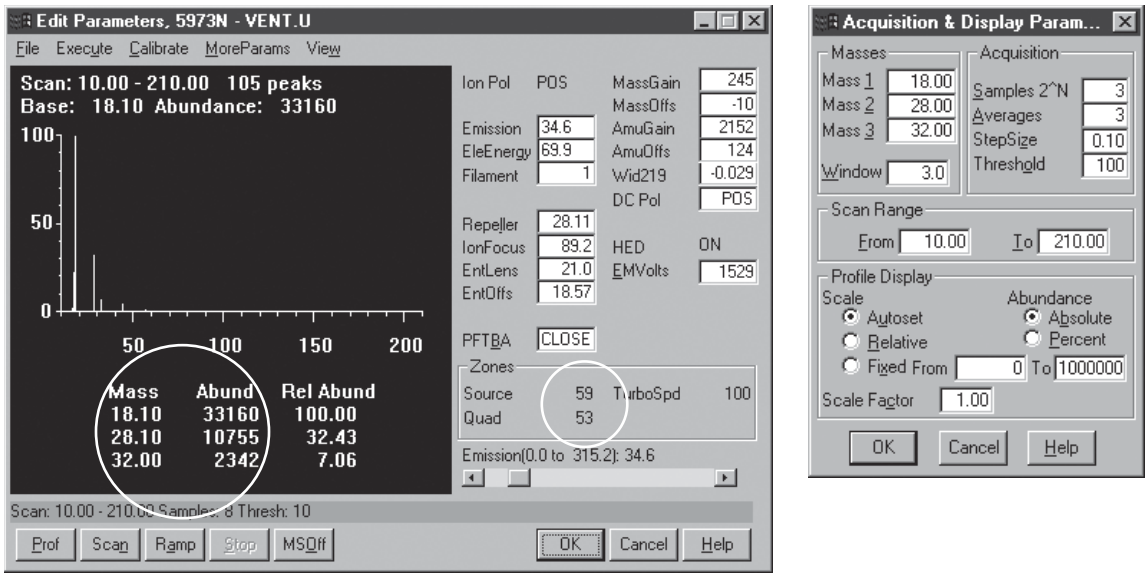


Figure 7. Pumpdown background checking.

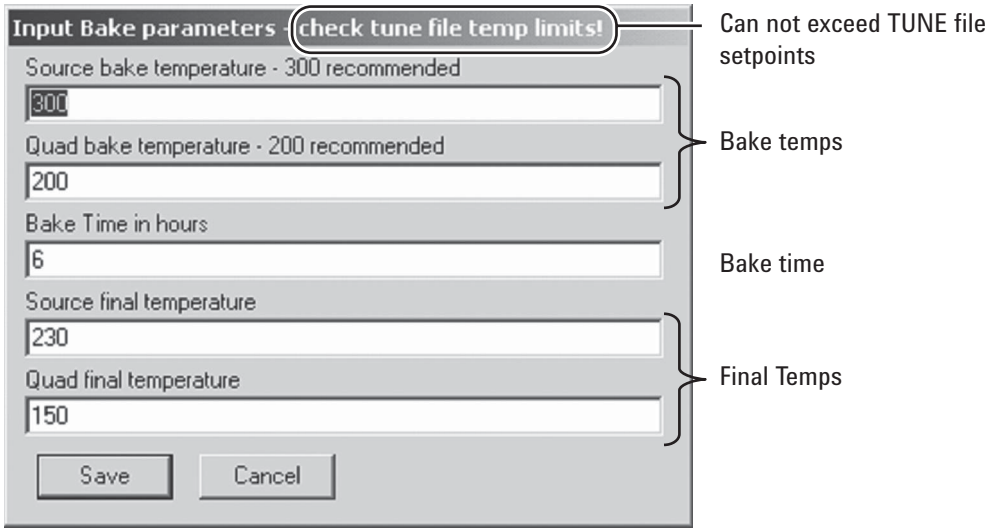


Figure 8. This is for manual bakeout of the source and quad. Add the file C:\MSDCHEM\MSEXE\Bake.mac Call as macro "bake.mac",go <enter>.

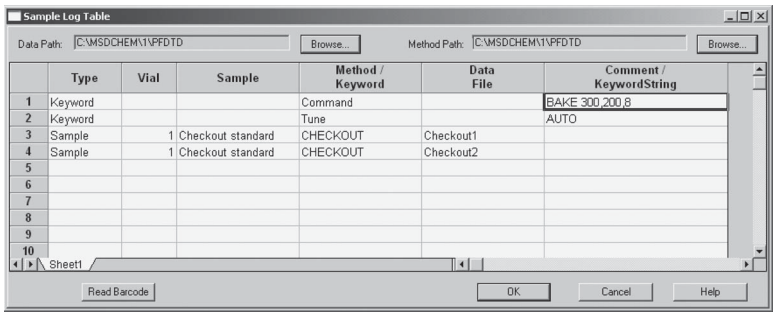
that shown in Figure 9. Line by line, this checkout sequence:

1. Calls the bake.mac and sets the baking temperatures of the ion source and quadrupole and the hours of bake time as BAKE SourceTemp, QuadrupoleTemp, Hours. Here the figure shows the source is baked at 300 °C and the quadrupole at 200 °C for 8 hours.
2. Source and quadrupole return to the temperatures of the loaded method tune file and then executes an AutoTune.
3. Injects a method performance standard twice.

The user can then examine the tune report for air and water and the datafiles of the performance standard to see that the instrument is operating within the required specifications of the user's analysis.

Instructions for Downloading the Macros from the Agilent Web Site

Go to http://www.chem.agilent.com/cag/servsup/softdocs/ssbMain.html?anch=MS. You must Register and acquire a username and a password. You also must have a valid MSD Productivity ChemStation registration number. Then go to the MSD



1. Calls Bake Macro
   Bake *SourceTemp, QuadTemp, Hours*
   This example sets source to 300 and quad to 200 for 8 hours
2. Runs AUTOTune
3-4. Checks Standards using the method CHECKOUT.M

Figure 9. Pumpdown sequence: Checkout.S.

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