

Mass Spectrometer General Maintenance Protocol

1 Scope

The purpose of this protocol is to provide general guidelines for maintenance of mass spectrometry (MS) instruments. This document applies to personnel using the associated instrument(s)/equipment in Quantico, VA in the following disciplines/categories of testing: Drug chemistry, toxicology, paint, explosives (chemistry), fire debris, and Chemistry Unit general physical and chemical analysis. Refer to the "General Instrument Maintenance Protocol" for overall instrument maintenance guidelines and definitions.

2 Principle

These instruments are obtained from several different manufacturers. All instruments eventually require maintenance, troubleshooting, and repair. Although the user interface and hardware fittings may differ, the overall instrument principles and maintenance are similar. The mass spectrometers are typically used in tandem with either a gas chromatograph (GC) or a liquid chromatograph (LC). Refer to the "Gas Chromatograph General Maintenance Protocol" and the "Liquid Chromatograph General Maintenance Protocol" for chromatographic system maintenance.

3 Equipment/Materials/Reagents

Any materials (such as pump oil and solvents) and all replacement parts will meet manufacturer's specifications and recommendations. Manufacturer's instrument manuals and specific performance monitoring protocols are generally the best source for this information.

4 Standards and Controls

All standards, solutions, and gases required are specified in the appropriate SOP.

5 Tuning

As defined in the "General Instrument Maintenance Protocol" tuning refers to the adjusting of parameters (e.g., lens voltages) to maximize instrument performance. All mass spectrometers provide a general automatic tune function. Individual instrument performance monitoring protocols have specific information on use of this function. Mass spectrometers also provide the ability to manually tune the same parameters. Manual tuning can be used as needed, provided that the required documentation is provided, and the decision criteria specified in the performance monitoring protocol is achieved.

6 Calibration

Any procedures used to calibrate and/or verify the integrity of the instrument will be specified in the appropriate SOP.

7 Sampling or Sample Selection

Not applicable.

8 Procedures

8.1 Preventative Maintenance

Each type and model of an instrument may have different, specialized components requiring specific preventative maintenance. Suggested step-by-step directions for specific maintenance procedures may be found in the manufacturer's instrument manuals. The following procedures are generic in nature and are included for reference.

8.1.1 Ion Volume Cleaning

Systems equipped with a removable ion volume should have the volume cleaned regularly. Lint-free gloves should be worn during the disassembly and reassembly of the mass spectrometer. Volume performance can be monitored by use of the performance monitoring standard criteria outlined in the instrument performance monitoring protocol. Although the interval is left to the operator, it is suggested that the volume at least be visually inspected on a daily basis. Volume-cleaning is the responsibility of all MS-trained operators.

- a. Remove the inner ion volume (with the filament and column inlet holes) from the outer housing.
- b. Mix a slurry of aluminum oxide and methanol.
- c. Thoroughly clean the inner and outer surfaces of both pieces of the volume with the slurry. Using a cotton-tipped applicator, clean all dark or discolored areas, particularly around holes.
- d. Place the ion volume parts in a beaker with deionized water and sonicate for one minute.
- e. Thoroughly rinse the parts with deionized water followed by methanol.
- f. Re-assemble the volume.

8.1.2 Vacuum Pumps

All MS systems have one or more rough/mechanical pumps. It is suggested that the pump oil level and clarity be checked yearly, and changed if needed. Some systems also have one or more turbo pumps as well. Turbo pumps are very sensitive and vary greatly, even within the same instrument. It is suggested that the oil not be replaced in turbo pumps. For changing the oil in the rough/mechanical pumps:

- a. Vent the MS.
- b. Allow the pump to cool for at least 10 minutes before continuing.
- c. Open the pump vent/fill hole.
- d. Place a sturdy plastic container under the oil drain.
- e. Open the oil drain and allow the old oil to empty.
- f. Add 10-20 mL of fresh oil to the pump with the drain open in order to flush the system.
- g. Replace the cover on the oil drain.
- h. Fill the pump with fresh oil until the proper fill level is noted in the level indicator.
- i. Replace the cover on the pump vent/fill hole.
- j. Seal, label and dispose of the used oil as outlined in the *FBI Laboratory Safety Manual*.
- k. Repeat for each vacuum pump on the system.

8.2 Corrective Maintenance

Each type and model of an instrument may have different, specialized components requiring specific corrective maintenance. Suggested step-by-step directions for specific procedures may be found in the corresponding manufacturer's instrument manuals. When performed, relevant corrective maintenance will be entered into the appropriate QA/QC logs. The following procedures are generic in nature and are included for reference.

8.2.1 Source Bake-Out

It should be noted that an occasional overnight baking-out of the detector may be useful when elevated baselines and other interferences are observed. The source temperature can be temporarily raised to 250°C. The transfer line should not be set to a temperature above the limit of the GC column. Please refer to specific column documentation for more information.

- a. Note the current source temperature.
- b. Set the source temperature to 250°C.
- c. Allow the source to bake-out for several hours.
- d. Return the source temperature to the original setting.
- e. Allow the source to cool to the original temperature before operating the instrument.
- f. This procedure can be repeated keeping the source temperature elevated overnight. However, if the problems persist, it is likely that the source and/or analyzer need to be cleaned.

8.2.2 Source Cleaning

The source will be cleaned on MS systems as needed, based on system performance. All systems require the removal of the entire source.

- a. Vent the MS system and turn off the main power.
- b. Allow the source to cool before continuing.
- c. Open the vacuum manifold.
- d. Disconnect any gas lines or electrical connections to the source.
- e. Loosen and/or remove source retaining bolts and clips.
- f. Remove the source.
- g. Disassemble the source in order to separate the lenses and any surfaces that come in contact with the ionization chamber.
- h. Mix a slurry of aluminum oxide and methanol.
- i. Thoroughly clean the pieces of the source with the slurry. Using a cotton-tipped applicator, clean all dark or discolored areas, particularly around holes. Warning: Only clean metal surfaces.
- j. Place the parts in a beaker with deionized water and sonicate for one minute.
- k. Thoroughly rinse the parts with deionized water followed by methanol.
- l. Re-assemble the source.

- m. Place the source in the manifold and secure.
- n. Reconnect all gas lines and electrical connections.
- o. Seal the manifold.
- p. Turn on the main power and pump down the system, observing for vacuum leaks.

8.2.3 Analyzer Cleaning

MS systems in the Quantico laboratory employ three types of analyzers: Ion trap, quadrupole, and time-of-flight. In general, these analyzers do not need regular cleaning. However, an ion trap can be easily cleaned when venting the system for source cleaning. A quadrupole is more sensitive to shock and manipulation, and should only be cleaned when warranted by poor performance. If the time-of-flight needs to be cleaned, contact appropriate instrument support personnel.

8.2.3.1 Cleaning an Ion Trap

- a. Vent the MS system and turn off the main power.
- b. Allow the analyzer to cool before continuing.
- c. Open the vacuum manifold.
- d. Disconnect any gas lines or electrical connections to the analyzer assembly.
- e. Remove the analyzer assembly from the manifold.
- f. Disassemble the ion trap and separate the ring and center electrodes.
- g. Mix a slurry of aluminum oxide and methanol.
- h. Thoroughly clean the surfaces of the electrodes using the slurry. Using a cotton-tipped applicator, clean all dark or discolored areas. Warning: Only clean metal surfaces.
- i. Place the parts in a beaker with deionized water and sonicate for one minute.
- j. Thoroughly rinse the parts with deionized water followed by methanol.
- k. Reassemble the ion trap and analyzer assembly.
- l. Place the analyzer assembly in the manifold and reconnect any gas lines and electrical connections.

- m. Seal the manifold.
- n. Turn on the main power and pump down the system, observing for vacuum leaks.

8.2.3.2 Cleaning a Quadrupole

- a. Vent the MS system and turn off the main power.
- b. Allow the analyzer to cool before continuing.
- b. Open the vacuum manifold.
- d. Disconnect any gas lines or electrical connections to the quadrupole.
- e. Remove the quadrupole from the manifold.
- f. Mix a slurry of aluminum oxide and methanol.
- g. Thoroughly clean the first inch of the inner surfaces of the quadrupole with the slurry. Using a cotton-tipped applicator, clean area of interest, particularly dark or discolored areas. Warning: only clean inner metal surfaces.
- h. Rinse by running water through the inside of the quadrupole for several minutes, until all aluminum oxide has been removed.
- i. Thoroughly rinse with methanol and allow quadrupole to dry.
- j. Place the quadrupole in the manifold and reconnect any gas lines and electrical connections.
- k. Seal the manifold.
- l. Turn on the main power and pump down the system, observing for vacuum leaks.

8.2.4 Filament Replacement

Generally, a broken filament results in a total loss of ions rather than degraded system performance. To replace a filament:

- a. Vent the MS system and turn off the main power.
- b. Allow the source to cool before continuing.
- c. Open the vacuum manifold.

- d. Disconnect any gas lines or electrical connections to the source.
- e. Loosen and/or remove source retaining bolts and clips.
- f. Remove the source.
- g. Disassemble the source in order to expose the filament.
- h. Unplug the old filament and replace it with a new one.
- i. Re-assemble the source.
- j. Place the source in the manifold and secure.
- k. Reconnect all gas lines and electrical connections.
- l. Seal the manifold.
- m. Turn on the main power and pump down the system, observing for vacuum leaks.

9 Instrumental Conditions

Refer to the appropriate procedures outlined in section 7, manufacturer's instrument manuals, or the instrument's performance monitoring protocol for specific instrumental conditions to be used during maintenance procedures.

10 Decision Criteria

Every performance monitoring protocol will have specific decision criteria to determine if the instrument is operating properly. If these should fail, the operator should refer to the 'Decision Criteria' section of the specific performance monitoring protocol. Additional information is provided in the 'Corrective Maintenance' section of the "General Instrument Maintenance Protocol".

11 Calculations

Not applicable.

12 Measurement Uncertainty

Not applicable.

13 Limitations

Only properly trained personnel will perform duties involved in the operation, maintenance, or troubleshooting of any instrument. Instrument-specific limitations will be specified in the appropriate SOP.

14 Safety

Take standard precautions for the handling of all chemicals, reagents, and standards. Refer to the *FBI Laboratory Safety Manual* for the proper handling and disposal of all chemicals. Personal protective equipment should be used when handling any chemical and when performing any type of analysis. Many instrument components are held at temperatures of 250°C and higher. Precautions should be taken to prevent the contact of skin with heated surfaces and areas.

15 References

Instrument Operation and Systems Support SOP Manual.

Manufacturer's Instrument Manuals for the specific models and accessories used.

"General Instrument Maintenance Protocol" (Inst 001) *Instrument Operation and Systems Support SOP Manual.*

"Gas Chromatograph General Maintenance Protocol" (Inst 002) *Instrument Operation and Systems Support SOP Manual.*

"Liquid Chromatograph General Maintenance Protocol" (Inst 003) *Instrument Operation and Systems Support SOP Manual.*

FBI Laboratory Safety Manual.

FBI Laboratory Quality Assurance Manual.

FBI Laboratory Operations Manual.

| Rev. # | Issue Date | History |
|--------|------------|---|
| 0 | 06/21/06 | New document which replaces original also titled "Mass Spectrometer General Maintenance Protocol." |
| 1 | 10/04/18 | Updated Section 1 Scope to include applicable disciplines/categories of testing. Changed 'Chemistry Unit' to 'Quantico laboratory' and added 'appropriate instrument support personnel' in Section 8.2.3. Updated abbreviation for IOSS in Section 15 and header. Updated section heading for Section 7 and updated wording in Section 10 for clarification |

Approval

Redacted - Signatures on File

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