1.0 PRINCIPLE:

GC with MS and Headspace analysis may be used in both qualitative and quantitative methods. Proper instrument maintenance is necessary to provide consistent, accurate and reliable results. The check solutions used are to verify acceptable working conditions on the day of use. This laboratory utilizes Hewlett-Packard 6890 gas chromatograph with 5973 mass selective detector and a Schimadzu GC 2010 with an AOC autosampler. Equipment used is the laboratory is safeguarded against unwarranted changes as stated in the GL-2 SOP. All comparison mass spectral libraries are individually identified, safeguarded, and controlled in the Toxicology section

Note: A particular instrument may be accepted for use in a particular situation with a "failed" parameter depending on the nature of the failure and the proposed use. (E.g. EM voltage failure in an instrument that will be used for cocaine/BE SIM analysis. Such a failure would not be expected to affect the validity of the analytical batch and therefore the instrument may, with appropriate annotation be used for that purpose. In contrast, the instrument would not be considered available for use in a drug "screen" protocol where sensitivity would be an issue.)

2.0 SAFETY:

As with any electrical device there is a chance of electrical shock if not handled properly, do not perform maintenance on this instrument unless trained to do so. Helium gas is used for the carrier gas note precautions for the handling of gas tanks. For further information refer to the instruments user's manual.

3.0 Procedure:

3.1 GCMS Daily check: A check list is utilized to indicate that specific items are performed daily prior to the instrument being marked as acceptable to use. (The Daily checks are documented on the GC/MS Daily Check sheet)

3.1.1 ATUNES and STUNES are run each day of use depending on the type of analysis (SIM or SCAN) needed.

3.1.2 The following parameters are reviewed;
Correct mass assignments (69,219 and 502)

Peak widths (acceptable range 0.45-0.65)
Relative ion abundances (69 at 100%, 219 >30% and 502 >4%)
Isotope masses (1AMU +/- 0.2 of parent peak)
Isotope ratios
70 (0.5%-1.5%); 219 (2%-8%); 503 (5%-15%)
EM voltages are recorded to help track the condition of the source.
(to perform tunes, refer to the HP5973 MSD Reference Collection)

3.1.3 Air and Water Checks: Performed daily to verify that there are no leaks in the GC system (refer to the HP5973 MSD Reference Collection). The ions corresponding to water m/z 18, nitrogen m/z 28 and carbon dioxide m/z 44 are checked to verify they are less then 10% of m/z 69 and that any trend does not indicate a leak.

3.1.4 Daily Standard Mix; (qualitative mixture):
This can include drug classes such as PCP, Barbiturates, Cocaine, CHEP, Opiates, Methadone, Phenethylamines such as MDMA, and Benzodiazepines. (the analytes can be changed as the need arises with the consultation of the section supervisor or Laboratory Director). This mix is run before any sample batches run that day to verify that there are no shifts in the retention times and that the fragmentation patterns are consistent.

The following parameters are reviewed:
- Non-target peaks (if present must be at an acceptable level or explainable, for example, the presence of a 6-MAM peak due to the breakdown of Heroin)
- All target peaks are present at appropriate levels.
- Retention time of CHEP (verify that no major unexplainable shifts have occurred)
- Abundance of CHEP (no major loss of sensitivity seen).
- CHEP spectral match is acceptable.

3.2 MAINTANENCE: Note: Reference material is HP5973 MSD Reference Collection; for specific guidance for required maintenance see this collection.

3.3 Daily Check (operational set-up):

3.3.1 Wash vials are emptied and refilled (one with chloroform and one with ethyl acetate other appropriate solvents are acceptable)

3.3.2 Helium tank pressure is checked (Tank should be changed when pressure declines below 300 psi.

3.3.3 Septa: the instrument septa should be changed approximately every 100-150 injections, and more frequently if and as indicated.

3.3.4 Liner: Changed as needed. Indications that the liner should be replaced include poor peak shape, extraneous peaks, loss of sensitivity.

3.3.5 Column: The front and/or rear of the column is clipped as needed. Indications that the column needs clipping include poor peak shape, extraneous peaks, loose of sensitivity.

Note: The column will be clipped or replaced as needed. Indications that the column needs replacement are a severe loss of sensitivity, poor peak resolution, excessive column bleed or extraneous peaks.

3.3.6 Filaments; the filaments are replaced as needed. There are two filaments per instrument, when one is no longer functional the instrument can be switched to use the second filament, when both filaments are no longer functional, they must be changed. When a filament is blown the instrument gives an error message and no data can be collected.

3.3.6 Gold seal; this is changed as needed as noted by extraneous peaks or loss of sensitivity.

3.3.7 Cleaning the Source; the source is cleaned as needed. An indication that the source needs to be cleaned is a high voltage multiplier value.

3.3.8 Changing oil for the rough pump; this is performed as needed the oil level is checked daily with a visual inspection for a leak.

3.3.9 Other Maintenance; performed as needed

3.4 GC Headspace Daily check: A check list is utilized to indicate that specific items are performed daily prior to the instrument being marked as acceptable to use. (The Daily checks are documented on the GCHS Daily Check sheet)

3.4.1 Daily calibration (multi pt. for Volatiles and GHB or Single pt.)

3.4.2 The type of Analysis performed
(Volatile, GHB, Other)

3.4.3 FID ignition check. Performed before each use to verify that Flame detector is in proper working order.

3.4.4 Tank gas pressure check:
Should be above 300 psi before daily run.

The following parameters are reviewed:
- Non-target peaks (if present must be at an acceptable level or explainable, for example, the presence of a 6-MAM peak due to the breakdown of Heroin)
- All target peaks are present at appropriate levels.
- Retention time of CHEP (verify that no major unexplainable shifts have occurred)
- Abundance of CHEP (no major loss of sensitivity seen).
- CHEP spectral match is acceptable.

3.4.5 The Daily check list shall include the Laboratory's unique instrument number with appropriate date any actions were taken.

3.5 Maintenance and Corrective action taken: Note: Reference material is Schimadzu Reference Collection; for specific guidance for required maintenance see this collection.

3.6 Daily Check (operational set-up):

3.6.1 Gas tank pressures is checked (Tanks should be changed when pressure declines below 300 psi.

3.6.2 Septa: the instrument septa should be changed approximately every 100-150 injections, and more frequently if and as indicated.

3.6.3 Liner: Changed as needed. Indications that the liner should be replaced include poor peak shape, extraneous peaks, loss of sensitivity.

3.6.4 Column: The front and/or rear of the column is clipped as needed. Indications that the column needs clipping include poor peak shape, extraneous peaks, loose of sensitivity.

Note: The column will be clipped or replaced as needed. Indications that the column needs replacement are a severe loss of sensitivity, poor peak resolution, excessive column bleed or extraneous peaks.

3.6.5 Other Maintenance; performed as needed

REFERENCES:

HP5973 MSD Reference Collection, discs 1-3; Feb. 1998 (Instrument tutorial)
Schimadzu operational collection discs.