1.0 PRINCIPLE:

Proper instrument performance is necessary to provide consistent, accurate and reliable results. To verify acceptable working conditions on the day of use, check solutions used with each instrument. This laboratory utilizes Enzyme Multiplied Immunoassay Techniques (EMIT), gas and liquid chromatography with mass selective detectors and head space gas chromatographs with Flame Ionization Detector (FID). Limited access to the Toxicology Laboratory safeguards the equipment against unwarranted changes. All comparison mass spectral libraries are individually identified, safeguarded, and controlled in the Toxicology unit

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. The reasoning to accept the instrument for use will be clearly noted in the maintenance logbook for that day.

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.

3.0 Procedure:

3.1 **GC/MS Day of Use Set-up**: A check list is utilized to indicate that specific items are performed each day of use prior to the instrument being marked as acceptable to use. (These checks are documented on the GC/MS Daily Check sheet)

3.1.1 ATUNES 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.

3.1.3 Air and Water Checks: Performed each day of use to verify that there are no leaks in the GC system. 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 than 10% of m/z 69 and that any trend does not indicate a leak. Note: for the Agilent 5975 and 5977 instruments the air and water check is part of the tune.

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 unit Lead or Deputy 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.

3.1.5 General Maintenance:

3.1.5.1 Wash vials are emptied and refilled (one with hexane and one With ethyl acetate other appropriate solvents are acceptable)

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

3.1.5.3 Septa: the instrument septa should be changed as needed.

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

sensitivity.

3.1.5.5 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.

3.1.5.6 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.

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

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

3.1.5.9 Changing oil for the rough pump; this is performed as needed the oil level is checked each day of use with a visual inspection for a leak.

3.1.5.10 Other Maintenance; performed as needed

3.2 **GC Headspace Day of Use Set-up**: A check list is utilized to indicate that specific items are performed each day of use prior to the instrument being marked as acceptable to use. (These checks are documented on the GC-HS Instrument Maintenance Log sheet; the instrument number will be included on the check sheet)

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

3.2.2 Tank gas pressure check: Should be above 300 psi before each day of use run.

3.2.3 Dy of Use calibration (multi point for Volatiles) – see TX-21 Volatiles for acceptance guidance.

3.2.4 Maintenance:

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

3.2.4.2 Septa: the instrument septa should be changed as needed.

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

3.2.4.4 Columns: 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.

3.2.4.5 Other Maintenance; performed as needed.

3.3 **LCMS Day of UseSet-up**: A check list is utilized to indicate that specific items are performed each day of use prior to the instrument being marked as acceptable for use. (These checks are documented on the LCMS pre-run checklist).

3.3.1 LCMS Day of Use Performance Check: A performance check solution, composed of neat reference standards, is analyzed prior to analyzing casework to verify proper instrument performance.

3.3.2 Performance Checks must meet the following criteria:

- Analyte peaks are present
- No unexpected analyte peaks
- Chromatography must be acceptable

- Criteria should be met for peak identification by software: proper retention time, correct ion ratios, sufficient responses to meet minimum peak area count

3.3.3 If the performance check fails take corrective action.

3.3.4 Corrective Actions: If the performance check does not meet the acceptance criteria check to make sure:

- The pumps are not “open” and thus directing effluent to waste.
- The correct column is installed and in the proper orientation.
- The correct method was opened in the software.

3.3.5 If there are no ions being generated it is likely that the desolvation line is clogged. Clean and/or replace the desolvation line if necessary.

3.3.6 Consult the unit Lead if instrument the check continues to fail.

3.4 **EMIT**: The EMIT is calibrated once per week or as per SOP TX-20.

3.4.1 Each day of use:

- Check the needle rinse and HCl bottles, these should be full.
- Check the waste bottles, empty if needed
- Fill the water reserve if needed
- Fill reagents, if needed

3.4.2 Periodic Maintenance

- Perform needle and rotor rinse (once every 7 days)
- Change Syringes
- Change Rotor

REFERENCES:

Schimadzu operational collection discs

Revision History

Revision 3	Removed reference to HP 5973 Reference collection. Changed reference of LCMSMS to LCMS (secion 3.3.1) to keep consistent with rest of section.