6.17 Gas Chromatography/Flame Ionization Detection (GC/FID) and Gas Chromatography/Mass Spectrometer (GC/MS)

The following section shall be used to ensure that the Gas Chromatography/Flame Ionization Detection (GC/FID) and Gas Chromatography/Mass Spectrometers (GC/MS) are properly maintained for accurate qualitative and/or quantitative analysis of case samples.

The Toxicology Unit currently uses Agilent gas chromatographs (models 6890 and 7890) and mass spectrometers (models 5973 and 5975). All of these systems utilize gas chromatography as the separation technique and flame ionization detection and/or quadrupole mass spectrometers as analyzers.

6.17.1 Maintenance

6.17.1.1 Before each run, the examiner shall:

- Fill wash bottles (see **6.17.3.2** "Note: This may need to be performed periodically to ensure sufficient wash bottle volume until completion of the run."). Documentation of who filled the bottles and date(s) this was done shall be included in the case file.
- Empty waste vials if needed.
- Evaluate and tune the mass spectrometer using the following methods:

6.17.1.1.1 Manual Leak Check

6.17.1.1.1.1 A manual leak check may be performed before an autotune. This is a specific measurement option available in the ChemStation software where water (18 m/z) and common air components such as nitrogen (28 m/z), carbon dioxide (44 m/z), etc. can be checked to ensure they are in low abundance. These values should be evaluated and compared to previous trends.

6.17.1.1.1.2 If the manual leak check results are consistent with established trends, the instrument should be autotuned.

6.17.1.1.1.3 If the manual leak check results are not consistent with established trends, it may be improved by tightening fittings, etc. If this problem cannot be resolved, the instrument shall be marked as "out of service" until repaired/replaced.

6.17.1.1.2 Autotune

6.17.1.1.2.1 An autotune (atune) shall be performed before each run. This is a specific adjustment option available in the ChemStation software where perfluorotributylamine (PFTBA) is used as the tuning solution. This feature

adjusts various parameters on the MS for the best response based upon current conditions.

6.17.1.1.2.2 If the tuning results pass established criteria as set forth in the Appendix section (Autotune Criteria), the instrument shall be available for casework. This autotune will be good for 24 hours or until the related batch is complete.

6.17.1.1.2.3 If tuning results fall outside the established criteria, it shall be corrected, if possible, and the instrument shall be re-tuned. If this problem cannot be resolved, the instrument shall be marked as "out of service" until the instrument is repaired/replaced.

6.17.1.1.3 The manual leak check (if performed previous to the autotune) and the autotune report shall be stored with the instrument in the maintenance notebook.

6.17.1.2 The mechanical pump oil conditions should be periodically monitored (e.g., level, color, etc.) and should be changed every six months or whenever needed.

6.17.1.3 As a part of preventative maintenance, the following should be evaluated before each run and done as needed: replace the liner (including o-ring), replace the gold seal and washer, change the septum, clip/replace the column, change the syringes, clean the source, refill the PFTBA vial, change the traps, etc.

6.17.1.4 All maintenance or repairs shall be recorded in the instrument's maintenance notebook and include at a minimum: the date of the maintenance or repair, the initials of the person performing the maintenance or repair, and a description of the type of maintenance or repair performed. All maintenance or repairs performed by a technical service representative shall be recorded in the same manner.

6.17.1.5 After maintenance or repair, a performance check shall be done by performing an autotune and/or analyzing calibrators and/or controls.

6.17.2 Data Evaluation

6.17.2.1 Instrument operation shall be verified by analyzing known calibrators and/or controls with each batch of unknown samples.

6.17.2.2 Chromatographic quality is evaluated in each sample. General guidelines are that peaks should be symmetrical, separate, and be resolved to the baseline where applicable. In circumstances where there is less than ideal chromatography (e.g., blood samples taken from a decomposed individual), the examiner shall make every effort to discriminate between coeluting peaks and poorly resolved compounds.

6.17.2.3 Capillary columns and temperature programs shall be used which are capable of resolving the analytes in question. The associated methods are designed to:

- Detect and differentiate between compounds of interest
- Minimize coelution of target compounds
- Minimize potential for carryover of previously analyzed compounds
- Provide a mass spectrum suitable for analysis of data (e.g., SIM or full scan mode, depending on the target analyte)

6.17.2.4 Instrumental data may be temporarily stored as data files on the appropriate instrument computer and, if possible, should be archived electronically via an external hard drive, recordable compact discs, etc. A hard copy will serve as documentation and shall remain within the case file.

6.17.3 Quality Assurance and Prevention of Carryover

6.17.3.1 The vial sequence shall be checked by the analyst or designee both prior to and after the injection of samples.

6.17.3.2 The wash bottles shall be filled with an adequate amount of solvent (well above the minimum solvent level line) to ensure that carryover does not occur. Documentation of who filled the bottles and date this was done shall be included in the case file. Note: This may need to be performed periodically to ensure sufficient wash bottle volume until completion of the run.

6.17.3.3 Syringes should be checked periodically for wear and blockage and replaced if necessary.

6.17.3.4 An extracted negative control shall be run immediately following each highest standard and immediately after the last case sample in each run to demonstrate that no carryover is present. This negative control shall be evaluated to the same extent as a case sample.

6.17.3.5 Sample drug concentrations greater than the highest calibration point could potentially be a source for carryover, therefore, the subsequent sample(s) shall be examined. Any of the examined samples suspected to contain a compound as a result of carryover will be rerun.