

TENNESSEE BUREAU OF INVESTIGATION

Forensic Services Division

Microanalysis Quality Assurance Manual

GC/MS Quality Assurance Policy

Quality Assurance for the Gas Chromatograph/Mass Spectrometers

1. Maintenance

1.1. Before each run, the examiner shall:

- Fill wash bottles. This may need to be performed periodically to ensure sufficient wash bottle volume until completion of the run.
- Empty waste vials if needed.
- Evaluate and tune the mass spectrometer using the following methods:

1.1.1. Manual Leak Check

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.

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

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.

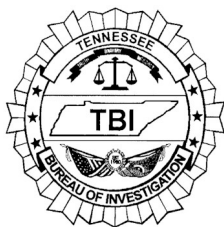
1.1.2. Autotune

1.1.2.1. An autotune 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.

1.1.2.2. If the tuning results pass established criteria, the instrument shall be available for casework. This autotune will be good for 24 hours or until the related batch is complete.

1.1.2.2.1. The three tuning masses should be within +/- 0.1 Atomic Mass Units (AMU) of 69.0, 219.0, and 502.0.

1.1.2.2.2. The peak widths should be 0.55 AMU +/- 0.1 AMU.



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1.1.2.2.3. Significant deviations in relative ratios of the tuning masses from previous tunes indicate instrumental problems and actions will be initiated in accordance with the instrument troubleshooting manual.

1.1.2.2.4. The electronic voltage settings should be close to previous tune values. Note: These voltages will gradually increase as the instrument ages and gets dirty. A significant change in one or several voltages could indicate a repair need.

1.1.2.2.5. The three masses 18, 28, and 32 need to be less than 20% of m/z 69.

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.

1.2. The manual leak check (if performed) and the autotune report shall be retained in the maintenance records for each instrument.

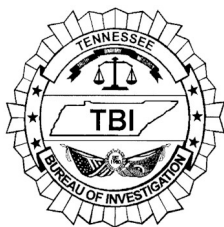
1.3. As a part of preventative maintenance, the following should be done monthly: autotune the mass spectrometer, analyze the column resolution check mixture (CRC), check inlet liner and O-ring and change as needed, check rough pump oil level and color and change as needed (this must be changed at least every six months).

1.4. As a part of preventative maintenance, the following should be evaluated periodically and done as needed: replace the gold seal and washer, change the Merlin Microseal, clip/replace the column, change the syringe, clean the source, refill the PFTBA vial, etc.

1.5. All maintenance or repairs shall be recorded in the instrument's maintenance records 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.

1.6. After maintenance or repair, a performance check shall be done by performing an autotune and analyzing the CRC.

2. Data Evaluation



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2.1. The resolution and sensitivity of the instrument shall be verified by analyzing a 1 μ L injection of the CRC at the beginning and end of each autosampler batch.

2.1.1. 1 mL of the commercially supplied Column Resolution Check Mixture will be dissolved in 9 mL of carbon disulfide (CS₂) to make a 10:1 dilution of the initial solution.

2.1.2. If the instrument does not detect the peak that represents decane, then the instrument failed the sensitivity portion of the test. If any of the peaks do not display baseline resolution, then the instrument failed the resolution portion of the test. The chromatogram will be maintained in each case file represented in the batch.

2.2. If the instrument is to be used in manual injection mode, a 1 μ L injection of the CRC will be analyzed once each day the instrument is used. The chromatogram will be evaluated as above and retained in each case file analyzed that day.

2.3. Chromatographic quality is evaluated in each sample. General guidelines are that peaks should be symmetrical, separate, and be resolved to the baseline when possible.

2.4. Instrumental data may be stored as data files on the appropriate instrument computer and, if possible, should be archived electronically in the TBI network. A printed copy will serve as documentation and shall remain within the case file.

3. Quality Assurance and Prevention of Carryover

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

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.

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

3.4. An appropriate blank shall be run in between each case sample in each run to demonstrate that no carryover is present.

3.5. Any of the examined samples suspected to contain a compound as a result of carryover will be rerun.