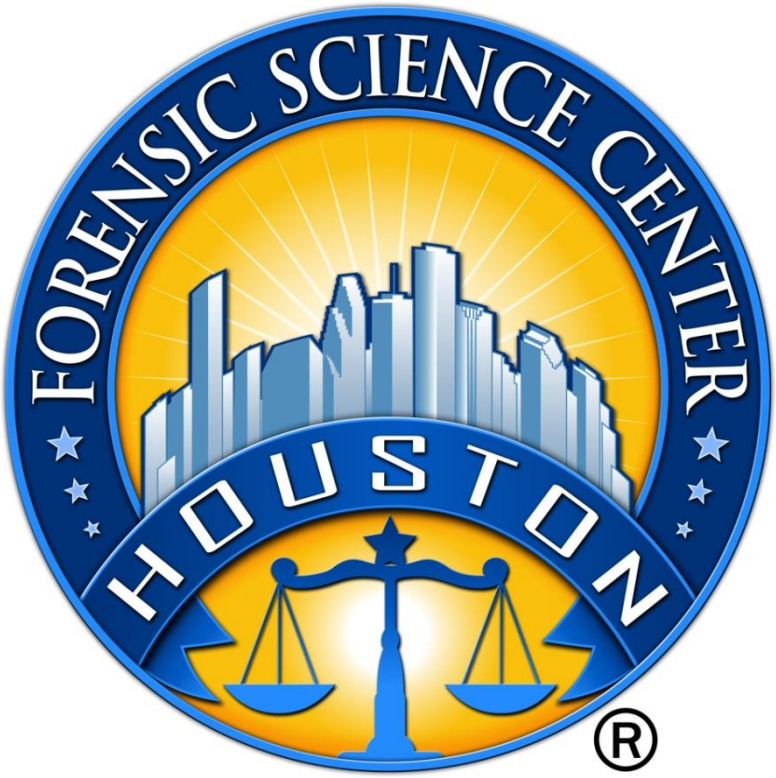


# Trace Analysis Section

# Gas Chromatography – Mass Spectrometry

Crime Scene/Digital and Multimedia Division

# 1. Standard Operating Procedure for the Use of Gas Chromatography-Mass Spectrometry (GC-MS) in the Trace Analysis Section.

## 1.1. Scope

1.1.1. This document covers the general operation, maintenance, and quality assurance/quality control of GC-MS instruments used for the analysis of trace evidence.

## 1.2. Safety

1.2.1. Use appropriate personal protective equipment.

1.2.2. Remove jewelry when performing instrument maintenance.

1.2.3. Normal GC-MS operating conditions include high temperatures and compressed gases.

1.2.4. Safety Data Sheets (SDS) are available in the Trace Section and may be accessed electronically. The content of the SDS should be reviewed prior to using a specific chemical.

## 1.3. Definitions:

1.3.1. PFTBA: PFTBA (perfluorotributylamine) is a standard utilized in Agilent mass spectrometers for auto-tuning the mass spectrometers, performing air and water checks, and for performing tune evaluations.

## 1.4. QA/QC

1.4.1. Performance Verification

1.4.1.1. The Mass Spectrometer (MS) is, at a minimum, tuned (referred to as an 'autotune' on Agilent GC-MS instruments) each month of use.

1.4.1.2. Autotuning specifications for Agilent GC-MS instruments:

1.4.1.2.1. The calibration solvent is PFTBA (perfluorotributylamine). The instrument uses three peaks from the spectrum of PFTBA to tune and calibrate the instrument. Those peaks are 69, 219, and 502.

1.4.1.2.2. The mass-to-charge base peak must be 69 or 219. The positions of masses 69, 219, and 502 must be within less than half a unit.

1.4.1.2.3. The isotope ratio of mass 70 to 69 must be between 0.5-1.6%. The isotope ratio of mass 220 to 219 must be between 3.2-5.4%. The isotope ratio of mass 503 to 502 must be between 7.9-12.3%.

1.4.1.2.4. The ratio of 219 to 69 must be greater than 40%. The ratio of 502 to 69 must be greater than 2.4%.

1.4.1.2.5. The mass 69 precursor must be less than or equal to 3%. The mass 219 precursor must be less than or equal to 6%. The mass 502 precursor must be less than or equal to 12%.

- To provide leak detection, the ratio of mass 18 (water) to 69 must be less than 20% and the ratio of mass 28 (nitrogen from air) to 69 must be less than 10%. To check if an Agilent GC-MS autotune is within the manufacturer's specifications, a tune evaluation may be run. Specifications are also listed in the instrument software.

1.4.2. Instrument Maintenance

1.4.2.1. Instruments are periodically maintained and their performance verified by running an autotune.

1.4.2.2. Maintenance events requiring an autotune include, but are not limited to: venting the source; changing columns; working on the inlet system; changing the carrier gas; replacing liners; maintenance services performed by an outside vendor; air water checks fail; or mass assignment fails a quality check. Changing a septum does not require autotuning.

1.4.2.3. If a performance problem is detected during use or routine maintenance, the instrument will be removed from service.

1.4.2.3.1. If a performance problem requires a service call, the Division Director or designee will be notified.

1.4.2.4. A record of all repairs and routine maintenance is kept in a log book near the instrument.

**1.5. Materials and equipment**

1.5.1. GC-MS

1.5.2. Methanol and other suitable organic solvents

1.5.3. GC-MS sample vials

1.5.4. High purity helium gas (compressed)

1.5.5. PFTBA

1.5.6. A chromatography column suitable to the application (usually a DB-1ms or DB-5ms, or equivalent)

1.5.7. Test tube or equivalent

**1.6. Procedure**

1.6.1. Sample Preparation

1.6.1.1. Sample preparation depends on the type of material being analyzed. If there is an SOP for the analysis of a specific type of material (such as fire debris or paint), refer to that document.

1.6.1.2. When analyzing unknown materials, as much information as possible should be obtained prior to analyzing the sample by GC-MS. Useful information includes solubility and pH (strongly acidic or basic materials should be extracted prior to analysis by GC-MS).

1.6.1.3. The following sample preparation procedure, often referred to as 'dilute and shoot', is usually successful; however, it may need to be modified according to case circumstances. Modifications must be verified using a known material.

1.6.1.3.1. Place a small amount of material in a test-tube or equivalent.

1.6.1.3.2. Add approximately 0.5 – 2 mL of an appropriate solvent (such as methanol).

1.6.1.3.3. If the sample is strong add a few drops to a GC-MS vial and fill with the same solvent; otherwise, add the mixture to a GC-MS vial.

1.6.1.3.4. Using an appropriate program analyze the sample by GC-MS.

1.6.1.3.5. A solvent blank is run prior to the sample.

1.6.1.3.6. A standard or standard test mixture shall be run each day of use prior to the analysis of casework to ensure the instrument is operating properly.

1.6.1.3.7. If a sample is too weak or nothing is detected during analysis, the sample can be concentrated using any of the following techniques or a combination thereof:

- A portion of the solvent can be evaporated
- A GC-MS injection volume can be increased
- If the GC-MS method uses a split injection, the split ratio can be decreased or the method modified to be run as splitless
- More of the unknown can be added to the solvent

1.6.1.3.8. If the sample is too strong it can be diluted by adding more solvent.

**1.7. Sample Analysis**

1.7.1. Chromatograms and mass spectra for samples, controls, and standards are either printed or stored electronically.

1.7.2. Peaks present in the GC-MS data are searched against databases and/or known standards.

1.7.3. A compound is identified when its mass spectrum closely matches that of a known compound. Compounds of interest found through computerized database searches are visually confirmed by the analyst. When possible, a standard is run using the same instrumental parameters. If no standard is available, the identity of the compound must be confirmed by comparing it to published data and by analyzing it using at least one other analytical technique.

1.7.4. When compounds are identified by GC-MS alone, their retention times and mass spectra are compared with standards, reference products, or reference materials. The retention time shall be within a 0.2-minute window and the data fragmentation shall correspond to standards or any other reference products or reference materials used.

**1.8. Method Development**

1.8.1. Minor changes to existing validated instrumental methods that do not affect the retention time or adversely affect peak shape are allowed.

1.8.1.1. Allowed minor changes to validated methods include:
- Manual injections
- Increasing or decreasing the injection volume
- Increasing or decreasing the split ratio
- Increasing or decreasing the AMU range
- Increasing or decreasing the run time

1.8.1.2. If a validated instrumental method is modified, the modified method is saved under a new name. These modified method changes are documented in the case record.

1.8.1.3. If the AMU range is modified for identification purposes, appropriate standards must also be run using the same modified AMU range. Modifications to the AMU range should only be made if deemed necessary after evaluating data from a validated instrumental method.

1.8.2. Changes to instrumental methods that affect the retention time or are needed for a new application may be required.

1.8.2.1. When a new instrumental method is required for the analysis of a new type of material (new to the Trace Analysis Section), the material is researched to determine if a method already exists in the literature or at another laboratory; or by evaluating the chemistry of the material and developing a method based on the analyst's previous knowledge of instrumental methods. The method shall be developed and tested using standards or commercial products of known composition.

1.8.2.2. When changes are made to an instrumental method that affect the retention time, a series of at least 5 injections of a standard will be run to show that the new retention time is reproducible.

1.8.3. Instrumental method development shall include the following steps:

1.8.3.1. The need for the new method shall be discussed with the Division Director or designee and approval (may be verbal), shall be obtained prior to commencing the work.

1.8.3.2. Once a satisfactory method is shown to work with standards or commercial products, it is printed and a printed copy kept in the methods log book by the instrument.

1.8.3.3. Instrumental method development may be part of a larger validation study. However, there may be the need to develop a new method for a specific case or new type of material.

1.8.3.4. A report documenting newly validated instrumental methods will be submitted to the Division Director or designee and to the Quality Division.

**1.9. New Instruments**

1.9.1. Performance verifications will be run on all new GC-MS instruments acquired by the Trace Analysis Section. Performance verification will include an Air and Water Check, Autotune or equivalent, an evaluation of the tune, running an applicable standard or mixture of standards, and a limit of detection study.

**1.10. References**

1.10.1. Silverstein, R.M., Bassler, G.C., Morrill, T.C., Spectrometric Identification of Organic Compounds, 4th Edition, John Wiley & Sons, New York, New York, 1981

1.10.2. Grob, R.L., Editor, Modern Practice of Gas Chromatography, 3rd Edition, John Wiley & Sons, Inc., New York, New York, 1995

1.10.3. McNair, H.M., Bonelli, E.J., Basic Gas Chromatography, 5th Edition, Varian Aerograph, Walnut Creek, California, 1968

1.10.4. Saferstein, R., Editor, Forensic Science Handbook, Volume II, 2nd Edition, Pearson Prentice Hall, Upper Saddle River, New York, 2005, Chapter 3, pp 81 – 109

1.10.5. Saferstein, R., Editor, Forensic Science Handbook, Volume I, 2nd Edition, Prentice Hall, Upper Saddle River, New York, 2002, Chapter 3, pp 117 – 159