FCS09 – SOP for Operating and Maintaining GC-MS and GC-FID Instruments

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1. Scope

1.1. This document establishes the procedures for instrument operation, preventative maintenance, and quality control that apply to instrumental analysis. The purpose of these maintenance and quality control (QC) procedures is to ensure that instruments are working properly and are free of contaminants before processing casework.

2. Background

2.1. To establish a procedure for regular instrument operation, maintenance, and QC to ensure quality and accuracy of reported casework results.

3. Safety

3.1. Reagent Toxicity:

3.1.1. Personnel should refer to the appropriate SDS for solvents and reagents used during analysis for any specific safety requirements.

3.1.2. For a complete review of required Health and Safety regulations of the Forensic Science Laboratory (FSL), see *DOM13 – DFS Health and Safety Manual*.

3.2. Protective Equipment:

3.2.1. Personnel should wear personal protective equipment (PPE) including: lab coat, gloves, and safety goggles when carrying out standard operating procedures.

3.2.2. Wear vinyl or nitrile gloves when handling these chemicals to prevent absorption through the skin. If any chemicals are spilled onto gloves, discard gloves into hazardous waste.

3.3. Training:

3.3.1. Formal training in use of instruments and software is necessary.

3.4. Personal Hygiene:

3.4.1. Universal Precautions must be followed. Care should be taken when handling chemicals or any biological specimen. Routine use of gloves and proper hand washing should be practiced.

3.4.2. Refer to DOM13 – DFS Health and Safety Manual.

3.5. Disposal of Waste:

3.5.1. Waste materials must be disposed of in compliance with laboratory, Federal, state, and local regulations. Solvents and reagents should always be disposed of in an appropriate container clearly marked for waste products and temporarily stored in a chemical fume hood.

3.5.2. Consult DFS Safety Officer for proper procedures.

4. Materials Required

4.1. Reagent Grade Acetonitrile (ACN) and Methanol (MeOH), Chloroform ($CHCl_3$), or higher purity.

4.2. Gas Chromatograph-Mass Spectrometer

4.2.1. Fully assembled, including: solvent vials, injection syringe, and other consumables.

4.2.2. Ultra-High Purity Helium tanks

4.2.3. Computer monitor equipped with instrumental software and attached printer.

4.3. Gas Chromatograph-Flame Ionization Detector (GC-FID)

4.3.1. Fully assembled, including: solvent vials, injection syringe, and other consumables.

4.3.2. Ultra-High Purity Helium tanks

4.3.3. Hydrogen Generator and water purification system

4.3.4. Computer monitor equipped with instrumental software and attached printer.

4.4. Binder/Folder for Autotune and Standard results, or electronic equivalent

4.5. Maintenance Logbook(s) and control chart(s)

5. Standards and Controls

5.1. Certified drug standard (Ex: Cocaine), or equivalent positive control

6. Calibration

6.1. Not applicable

7. Procedures

7.1. Gas Chromatography-Mass Spectrometry (GC-MS)

7.1.1. Daily Maintenance Procedure

7.1.1.1. Check the levels of the carrier gas source and record in the Gas Tank Usage Log (Document Control Number 30397)

7.1.1.2. Change gas tank source when empty. Replace helium tanks, update gas signage, and update logbook as appropriate.

7.1.1.3. Prior to each run, check wash solvent levels in the injector turret. Change or fill as necessary.

7.1.2. Weekly Maintenance Procedures

7.1.2.1. Weekly maintenance must be carried out each week on each instrument used for casework. If only one instrument is used for casework, only the instrument being used must undergo this weekly maintenance.

7.1.2.2. Each Monday is considered the start of a new week for weekly maintenance purposes (or the following business day if Monday is a holiday.)

7.1.2.3. Solvent Vials

7.1.2.3.1. Empty the waste vials in the injector turret.

7.1.2.3.2. Empty and refill Acetonitrile and/or Methanol in the wash solvent vials in the injector turret.

7.1.2.3.3. Empty and refill Acetonitrile, Methanol, and/or Chloroform in the solvent blank vials in the sample tray.

7.1.2.4. Syringe Cleaning

7.1.2.4.1. Clean autoinjector syringe using Acetonitrile and/or Methanol.

7.1.2.5. Run Autotune

7.1.2.5.1. Tune with Perfluorotributylamine (PFTBA) using Autotune function in ChemStation using the stune method.

7.1.2.5.2. Check autotune for excessive peaks, good tune peak shape, and water and oxygen levels under 10%.

7.1.2.6. Sequence

7.1.2.6.1. Save Sequence from the previous week.

7.1.2.6.2. File the previous week's sequence into the GC-MS logbook, or electronic equivalent

7.1.2.6.3. Load the Weekly Sequence template file and save as a new sequence using the current date with the format YYYY.MM.DD (Ex: 2015.12.25). The weekly sequence template shall contain all required runs as specified for a performance check.

7.1.2.7. Performance Check

7.1.2.7.1. Run a negative control (blank run) in the BLANK.M or other appropriate validated method, to

ensure that there are no contaminants present in the system.

7.1.2.7.2. If contaminants are present, additional blanks shall be run until a clean blank is obtained.

7.1.2.7.3. If a clean blank cannot be obtained, the instrument shall be taken out of service until the cause of contamination is found and removed.

7.1.2.7.4. Run a positive control (Cocaine or other standard run) to ensure the system is functioning properly.

7.1.2.7.5. The positive control must clearly display the peaks of interest with no significant background.

7.1.2.7.6. File the standard run and the blank before it in the Standards Logbook.

7.1.3. Monthly Maintenance Procedures

7.1.3.1. Change liner and septum

7.1.3.2. Replace solvent blank vials for Acetonitrile, Methanol, and/or Chloroform.

7.1.4. As-Needed Maintenance Procedures

7.1.4.1. A performance check must also be completed along with an autotune after any major maintenance that may modify the function of the equipment (i.e., changing of major parts, column trimming, venting, source cleaning). A new sequence shall be created in this event.

7.1.4.2. For minor service interruptions that do not require instrument venting or opening/changing instrument parts, a performance check shall be performed within the same sequence.

7.1.4.3. Other as-needed maintenance:

7.1.4.3.1. Clean MS ion source

7.1.4.3.2. Switch MS filament in use

7.1.4.3.3. Cut/Replace Column

7.1.4.3.4. Change liner and/or septum

7.1.4.3.5. Rinse septum port with solvent

7.1.5. Preventive Maintenance Schedule

7.1.5.1. All GC-MS instruments shall undergo an annual (i.e., once per calendar year) preventive maintenance by the vendor.

7.1.6. Control Chart and Logbook Maintenance

7.1.6.1. The significant parameters appropriate for the identification Cocaine (or other substance used for quality control) shall be recorded in the laboratory control chart for GC-MS when a performance check is completed. These parameters include:

7.1.6.1.1. If the blank is clear and whether or not it shows all expected peaks for standard.

7.1.6.1.2. Retention time and abundance of the analyte peak(s).

7.1.6.1.3. Peak heights for three characteristic m/z peaks

7.1.6.1.4. Noise abundance to calculate S/N ratio

7.1.6.2. The significant parameters appropriate for evaluation of MS function shall be recorded in the laboratory control chart for GC-MS when an autotune is completed. These parameters include:

7.1.6.2.1. Peak width

7.1.6.2.2. EMVolts

7.1.6.2.3. Oxygen level

7.1.6.2.4. Water level

7.1.6.3. A GC-MS maintenance log (Document Control Number 30398) shall be maintained for each instrument to reflect any maintenance procedures performed (along with corresponding dates and analysts) and operating status (i.e. online or offline).

7.1.7. Quality Control Procedures

7.1.7.1. A negative control (blank run) shall be run prior to each sample using the BLANK.M method, or the same method being used for analysis. The blank must be free of discernible peaks to be acceptable.

7.1.7.1.1. Note: If a minor peak is observed in a blank, the chemist may review and acknowledge their data to verify that the peak is an expected solvent peak, a result of column bleed, or minor artifact peak and contains no analytes of interest.

7.1.7.2. Sample run printouts shall be accompanied by the previous blank's printout to ensure the system was free of contaminants prior to the run of interest.

7.1.7.3. Each sample and standard vial must be labeled with unique identifying marks to ensure that the correct sample or standard is run.

7.1.7.4. When a run is complete, the vial number listed on the printouts should be compared to the corresponding vial in the autosampler tray to ensure that the correct sample is run.

7.1.7.5. Standard Runs

7.1.7.5.1. In order to conclusively identify a substance in a sample, a pure standard corresponding to the analyte of interest must be run during the same week as the sample and must be listed on the same sequence file as the sample.

7.1.7.5.2. A separate standard will be run for each analyte of interest being reported, but multiple samples may be matched up with the same standard.

7.1.7.5.3. A blank run must be performed before the standard as if it were a sample.

7.1.7.5.4. The retention time of the sample must match the retention time of the standard, meeting acceptance criteria defined in 7.1.7.8. Because concentrations of analytes can affect their retention times, if the retention time is outside this range, the sample or standard may be diluted with corresponding solvent, or concentrated and rerun.

7.1.7.5.5. Both the sample and standard must have similar mass spectra, meeting acceptance criteria listed in 7.1.7.8. When possible, both spectra shall also match a result in a MS reference library.

7.1.7.6. A copy of the matching standard printout and the blank run immediately before it shall be included with the printout of the sample.

7.1.7.7. A copy of the printout for each standard used to match casework and the blank run immediately before it shall be retained in the Standards Logbook.

7.1.7.8. GC-MS acceptance criteria

	Acceptance Criteria	Detail
GC PARAMETERS (GC-MS)	**Retention Time**	Retention time of analyte peak must match within 2% of standard or within 0.05 minutes, whichever is higher.
	S/N* Cut Off	Analyte TIC** must be more than three (3) times greater than noise.
	Peak width resolution	Analyte peak must be base peak resolved, as evaluated by the analyst.

*S/N = Signal-to-Noise Ratio; **TIC = Total Ion Chromatogram

	Acceptance Criteria	Detail
MS PARAMETERS (GC-MS)	**Peak number**	At least two (preferred three) characteristic peaks.
	Relative peak intensity	Analyte peak relative intensity shall match their reference standards by ±15%.
	Peak height	Characteristic peak signal intensity must be at least 500 a.u.*

*a.u. = arbitrary units

7.2. Gas Chromatography-Flame Ionization Detection (GC-FID)

7.2.1. Daily Maintenance Procedure

7.2.1.1. Check the levels of the carrier gas source and record in the Gas Tank Usage Log (Document Control Number 30397)

7.2.1.2. Change gas tank source when empty. Replace helium tanks, update gas signage, and update logbook as appropriate.

7.2.1.3. Prior to each run, check wash solvent levels in the injector turret. Change or fill as necessary.

7.2.1.4. Check hydrogen generator water level. Add water when necessary.

7.2.1.4.1. Note: Water must have a resistance greater than 18.2 MΩxcm, obtained from a water purification system.

7.2.2. Weekly Maintenance Procedures

7.2.2.1. Weekly Maintenance must be carried out each week on each instrument used for casework and must be performed prior to any other casework. If only one instrument is used for casework, only the instrument being used must undergo this weekly maintenance.

7.2.2.2. Each Monday is considered the start of a new week for weekly maintenance purposes (or the following business day if Monday is a holiday).

7.2.2.3. Solvent Vials

7.2.2.3.1. Empty the waste vials in the injector turret.

7.2.2.3.2. Empty and replace Acetonitrile and/or Methanol in the wash solvent vials in the injector turret.

7.2.2.3.3. Empty and replace Acetonitrile, Methanol and/or Chloroform in the solvent blank vials in the sample tray.

7.2.2.4. Syringe Cleaning

7.2.2.4.1. Clean autoinjector syringe using Acetonitrile and/or Methanol.

7.2.2.5. Sequence

7.2.2.5.1. Save Sequence from the previous week.

7.2.2.5.2. File the previous week's sequence in the GC-FID Logbook, or electronic equivalent.

7.2.2.5.3. Load the Weekly Sequence template file and save as a new sequence using the current date with the format YYYY.MM.DD (Ex: 2015.12.25). The weekly sequence template shall contain all required runs as specified for a performance check.

7.2.2.6. Performance Check

7.2.2.6.1. Run a negative control (blank run) in the SCRN165.M method. or other appropriate validated method. to ensure that there are no contaminants present in the system.

7.2.2.6.2. If contaminants are present, additional blanks shall be run until a clean blank is obtained.

7.2.2.6.3. If a clean blank cannot be obtained, the instrument shall be taken out of service until the cause of contamination is found and removed.

7.2.2.6.4. Run a positive control (GC Mix or other Standard run) to ensure the system is functioning properly.

7.2.2.6.5. The positive control must clearly display the peaks of interest with no significant background.

7.2.2.6.6. File the standard run and the blank before it in the GC-FID Logbook.

7.2.3. Monthly Maintenance Procedures

7.2.3.1. Change liner and septum

7.2.3.2. Replace solvent blank vials for Acetonitrile, Methanol, and/or Chloroform.

7.2.4. As-Needed Maintenance Procedures

7.2.4.1. A performance check must also be completed after any major maintenance that may modify the function of the equipment (i.e., changing of major parts or column trimming). A new sequence shall be created in this event.

7.2.4.2. For minor service interruptions that do not require opening/changing instrument parts, a performance check shall be performed within the same sequence.

7.2.4.3. Other as-needed maintenance:

7.2.4.3.1. Cut/replace column

7.2.4.3.2. Change liner and/or septum

7.2.4.3.3. Replace jet

7.2.4.3.4. Rinse septum port with solvent

7.2.5. Preventative Maintenance Schedule

7.2.5.1. All GC-FID instruments shall undergo an annual (i.e., once per calendar year) preventive maintenance by the vendor.

7.2.6. Control Chart and Logbook Maintenance

7.2.6.1. The significant parameters appropriate for the identification of individual substances or mixtures of heroin, cocaine, etc. (or other substance used for quality control) shall be recorded in the laboratory control chart for GC-FID when a performance check is completed. These parameters include:

7.2.6.1.1. If the blank is clear and whether or not it shows all expected peaks for standard

7.2.6.1.2. Retention time of the analyte peaks(s)

7.2.6.2. A GC-FID maintenance log (Document Control Number 30398) shall be maintained for each instrument to reflect any maintenance procedures performed (along with corresponding dates and analysts) and operating status (i.e. online or offline).

7.2.7. Quality Control Procedure

7.2.7.1. A negative control (blank run) shall be run prior to each sample using the BLANK.M method or the same method used in analysis. The blank must be free of discernible peaks to be acceptable.

7.2.7.1.1. Note: If a minor peak is observed in a blank, the chemist may review and acknowledge their data to verify that the peak is an expected solvent peak, a results of column bleed, or minor artifact peak that does not interfere with analytes of interest.

7.2.7.2. Sample run printouts shall be accompanied by the previous blank's printout in order to ensure the system was free of contaminants prior to the run of interest.

7.2.7.3. Each sample and standard vial must be labeled with unique identifying marks to ensure that the correct sample or standard is run.

7.2.7.4. When a run is complete, the vial number listed on the printouts should be compared to the corresponding vial in the autosampler tray to ensure that the correct sample is run.

7.2.7.5. Standard Runs

7.2.7.5.1. In order to identify a substance in a sample, a pure standard corresponding to the analyte of interest must be run during the same week as the sample and must be listed on the same sequence file as the sample. The substance must be run on the same method as the standard. For confirmation, this method shall be used in conjunction with a SWGDRUG Category A test, ASTM E2329-17, of which results shall be in agreement.

7.2.7.5.2. A separate standard shall be run for each analyte of interest being reported, but multiple samples may be matched up with the same standard.

7.2.7.5.3. A blank run must be performed before the standard as if it were a sample.

7.2.7.5.4. The retention time of the sample must match the retention time of the standard, meeting acceptance criteria defined in 7.2.7.8. Because

concentrations of analytes can affect their retention times, if the retention time is outside this range, the sample or standard may be diluted with corresponding solvent, or concentrated and rerun.

7.2.7.6. A copy of the matching standard printout and the blank run immediately before it shall be included with the printout of the sample.

7.2.7.7. A copy of the printout for each standard used to match casework and the blank run immediately before it shall be retained in the Standards Logbook.

7.2.7.8. GC-FID acceptance criteria

	Acceptance Criteria	Detail
GC-FID PARAMETERS	**Retention Time**	Retention time of analyte peak must match within 2% of standard or within 0.05 minutes, whichever is higher.
	S/N* Cut Off	Analyte signal intensity must be more than three (3) times greater than noise.
	Peak width resolution	Analyte peak must be base peak resolved, as evaluated by the analyst.

*S/N = Signal-to-Noise Ratio

8. Sampling

8.1. See *FCS02 – SOP for General Laboratory Procedures for FCU*

9. Calculations

9.1. Not applicable

10. Uncertainty of Measurement

10.1. See *FCS21 – Procedure for Uncertainty in Measurement.*

11. Limitations

11.1. Not applicable

12. Documentation

12.1. FCU Gas Tank Usage Log (Document Control Number 30397)

12.2. Maintenance Logbooks (Document Control Number 30398) and Control Charts

13. References

13.1. DFS Departmental Operations Manuals, (current revisions).

13.2. Forensic Chemistry Unit Quality Assurance Manual, (current revisions)

13.3. Forensic Chemistry Unit SOPs, (current revisions).

13.4. ASTM E2329-17, Standard Practice for Identification of Seized Drugs.