Deviation Request Form (DRF)

Directions: The Initiator will complete Sections A through C. Additional continuation pages can be included if necessary.

Initiator	Amanda Venable	**Date**	5/14/2021

A. Requested deviation applies to (Technical Procedure – include specific section):

Technical Procedure Gas Chromatography/Mass Spectrometry (GC-MS) - Version 14

See attached.

B. Requested deviation:

See attached.

C. Necessity for the deviation:

See attached.

D. Technical review and Authorization (to be completed by the Quality Manager and/or Technical Leader)
Comments(to include merits and impacts):

See attached.

Approved	☑	Yes	☐	No	Duration	1 year, or until next procedure revision
Signature	Amanda Battin Venable — Digitally signed by Amanda Battin Venable Date: 2021.05.17 11:24:30 -04'00'				Date	05-17-2021

E. Quality Assurance Authorization (to be completed by the Quality Manager, Forensic Scientist Manager or designee)

Acceptable within general QA guidelines and good laboratory practice?	☑	Yes	☐	No
Significant negative impact to Crime Laboratory Quality System?	☐	Yes	☑	No

Restrictions/limitations:

☑	Authorized	☐	Rejected	Signature	Katy Schell — Digitally signed by Katy Schell DN: cn=Katy Schell, o=North Carolina State Crime Laboratory, ou=Drug Chemistry Section, email=Kschell@ncdoj.gov, c=US Date: 2021.05.17 12:15:06 -04'00'	Date	5/17/2021

Form approved for use by: Timothy G. Suggs — Digitally signed by Timothy G. Suggs DN: cn=Timothy G. Suggs, o=North Carolina State Crime Laboratory, ou=Quality Manager, email=tsuggs@ncdoj.gov, c=US Date: 2014.12.19 09:01:52 -05'00'

A. Requested deviation applies to (Technical Procedure – include specific section)

Technical Procedure Gas Chromatography/Mass Spectrometry (GC-MS) - Version 14

5.7.5 For sample runs, the case record shall contain:

- Total Ion Chromatogram (TIC) for the corresponding blank.
- Total Ion Chromatogram (TIC) for the sample.
- Mass spectra of peaks of interest, such as common diluents, artifacts, or peaks comparable in size to any controlled substances present.
- Expanded mass spectra of phenethylamines and other compounds as needed
- Mass spectra of any reference material standards used for identification and its unique identifier, and the corresponding library search.
- When retention time is required for identification of a compound, the blank and standard TIC as well as the mass spectrum of the reference material shall be included. Retention times shall be indicated for comparison purposes.

B. Requested deviation:

5.7.5 For sample runs, the case record shall contain:

- Total Ion Chromatogram (TIC) for the corresponding blank.
- Total Ion Chromatogram (TIC) for the sample.
- Mass spectra of peaks of interest, such as common diluents, artifacts, or peaks comparable in size to any controlled substances present.
    - For items utilizing the hypergeometric sampling plan or threshold sample selection, mass spectra of the identified controlled substance(s) is required for each unit analyzed, however mass spectra of peaks of interest that are consistent between all units analyzed, are only required to be included once.
- Expanded mass spectra of phenethylamines and other compounds as needed
- Mass spectra of any reference material standards used for identification and its unique identifier, and the corresponding library search.
- When retention time is required for identification of a compound, the blank and standard TIC as well as the mass spectrum of the reference material shall be included. Retention times shall be indicated for comparison purposes.

C. Necessity for the deviation:

To prevent inclusion of redundant information into the case file and to streamline data processing in specific instances.

D. Technical review and Authorization (to be completed by the Quality Manager and/or Technical Leader). Comments (to include merits and impacts):

The exclusion of mass spectral data in the casefile for replicate peaks of interest for items utilizing the hypergeometric sampling plan or the threshold sample selection will not affect the quality of the case. All data generated from each unit will be evaluated and/or compared by the analyst during data processing. Furthermore, the associated mass spectrum for each substance being identified will be included for all units.

Technical Procedure for Drug Chemistry Gas Chromatograph/Mass Spectrometry (GC-MS)

1.0 Purpose - This procedure specifies the required elements for the calibration and use of the Agilent 6890 GC (or equivalent) interfaced to the Agilent 5973 Series MSD (or equivalent) for Drug Chemistry analyses.

2.0 Scope - This procedure applies to all GC-MS instruments used for drug chemistry analyses in the Drug Chemistry Sections of the State Crime Laboratory.

3.0 Definitions

- **Performance verification -** The initial confirmation of the reliability of a previously or externally validated method or instrument.
- **Probability Based Matching** – An algorithm designed to compare an unknown mass spectrum against a reference collection of mass spectra for the purpose of identification.
- **Quality control (QC) check -** Periodic confirmation of the reliability of equipment, instrumentation, and/or reagents.
- **Primary reference material –** Any reference material obtained from a source other than the State Crime Laboratory and which has documentation issued by the provider authenticating its chemical composition.
- **Secondary reference material –** Any reference material used in the course of casework that has its chemical composition verified by a primary reference material.
- **Diagnostic Ion** – A MS fragment ion that has structural relevance to the targeted analyte.

4.0 Equipment, Materials and Reagents

4.1 Equipment

- Agilent Gas Chromatograph 6890 Series (GC), or equivalent
- Agilent 5973 Series Mass Selective Detector (MSD), or equivalent
- Agilent Automatic Liquid Sampler
- PC with Agilent Analytical MSD Productivity ChemStation Software or equivalent
- Computer Printer or other data output device

4.2 Materials

- Sample vials, inserts, and caps
- 10 µL syringe
- A non-polar capillary column with a (5 %-Phenyl)-methylpolysiloxane stationary phase, such as a DB-5MS or RTx-5MS, or other column as needed
- Septum
- Liners
- Pump Oil
- Gold Seal

4.3 Commercial Reagents

- Methanol
- Hexane
- Chloroform

- Acetonitrile
- Ethyl acetate
- Helium gas, Grade 5.0
- Perfluorotributylamine [PFTBA], neat
- Acetone
- Methylene chloride
- Isopropyl alcohol

***Solvents listed above shall be ACS grade or higher**

4.4 Reference Materials

- Multi-component drug solutions
- Primary or Secondary reference material

5.0 Procedure

5.1 Instrument Performance Verification for New Instrumentation

5.1.1 New GC-MS instruments shall be installed by a manufacturer representative and shown to meet any manufacturer's requirements.

5.1.2 The GC-MS Coordinator or designee shall conduct a performance verification on new GC-MS instruments prior to use for casework.

5.1.2.1 The performance verification shall include successful tunes (see **5.4**) on three separate days.

5.1.2.2 The performance verification shall include the successful analysis of multi-component reference material standard solutions from **5.3.2** run on three separate days.

5.1.2.2.1 The mass spectra of each component shall be successfully compared to reference material as set forth in **5.7.3.**

5.1.2.2.2 The difference of the highest and lowest retention times of each component shall satisfy the criteria set forth in **5.3.2.3.**

5.1.2.3 The data shall be filed and maintained by the GC-MS Coordinator or designee to document set up of the new instrument.

5.1.2.4 A new entry for the instrument shall be made in the Resource Manager section of FA prior to use in casework. The new entry will include:

5.1.2.4.1 The manufacturer's serial number.

5.1.2.4.2 The unique section identifier for the new instrument.

5.1.2.4.3 A notation under "Verification Date" to reflect the date the performance verification was completed.

5.1.3 Each GC-MS instrument shall have a GC-MS Logbook, which shall be maintained electronically. The GC-MS Logbook shall consist of the Activity Log, the Maintenance Log, and Retention Time Log.

5.1.3.1 The Activity Log shall include the date, sample identifier, initials of operator, GC-MS method used, and comments for each sample analyzed. The Activity Log shall also include substances observed in the sample.

5.1.3.2 Any unusual error messages shall be recorded in the Activity Log.

5.1.3.3 If samples are rerun for any reason, a new entry shall be recorded in the Activity Log. (Blank solvent runs do not need to be recorded.)

5.1.3.4 The Activity Log shall contain the appropriate file name(s) for monthly performance check data. Other retention time reference material data may also be stored in the logbook (see **5.3.2.2**).

5.1.3.5 The Maintenance Log shall include the date, description of work performed, length of any column trimmed, parts replaced, and the initials of the person performing or documenting the maintenance.

5.1.3.6 Tunes shall be documented in the Activity Log, as described in **5.4.4**. The tune reports shall be stored electronically. Tunes performed to check instrument performance during maintenance or troubleshooting need not be retained.

5.1.3.7 All information stored in the GC-MS Logbook shall be archived at least annually into the FA object repository ("Manage Files") associated with each instrument.

5.2 Maintenance

5.2.1 Record maintenance at the time it is performed. The GC-MS Coordinator or designee shall file all maintenance records in the object repository ("Manage Files") for the specific instrument in the Resource Manager section of FA.

5.2.1.1 Document completion of maintenance in the Maintenance Log of the GC-MS Logbook associated with the specific GC-MS instrument.

5.2.1.1.1 If maintenance is performed over the course of several days, only one Action History event encompassing all work done needs to be recorded.

5.2.1.2 The GC-MS Coordinator or designee shall update the GC-MS Logbook when the instrument is placed in or out of service.

5.2.2 Record lengths of column trimmed in the Maintenance Log. A highlighted entry shall also be made in the Activity Log, documenting the column trimming. If the column is trimmed, the instrument shall be out of service until a monthly performance check is successfully completed (see **5.3.2**).

5.2.2.1 Standards run prior to the column maintenance shall not be used for retention time comparison after the column maintenance.

5.2.3 **Routine maintenance** – The routine maintenance schedule is a suggested minimum guideline. The maintenance schedule will be determined by the GC-MS Coordinator or designee based upon instrument use and performance.

5.2.3.1 Wash Vials

- Rinse and/or fill with the appropriate wash solvent daily when in use.
- Post-maintenance check: None.

5.2.3.2 Septum

- Replace at least monthly.
- Post-maintenance check: Successful tune (see **5.4**) followed by a successful blank as outlined in **5.3.3.3**.

5.2.3.3 Syringe

- Inspect monthly for cleanliness and ease of movement. Replace as needed.
- Post-maintenance check, if syringe is replaced: Successful tune (see **5.4**) followed by a successful blank as outlined in **5.3.3.3,** as well as a monthly performance check (see **5.3.2**).

5.2.3.4 Liner

- Replace as needed, or every six months.
- Post-maintenance check: Successful tune (see **5.4**) followed by a successful blank as outlined in **5.3.3.3,** as well as a monthly performance check (see **5.3.2**).

5.2.3.5 Pump Oil

- Change at least twice a year.
- Post-maintenance check: Successful tune (see **5.4**) followed by a successful blank as outlined in **5.3.3.3**.

5.2.3.6 Clean Source

- Clean at least annually.
- Post-maintenance check: Successful tune (see **5.4**) followed by a successful blank as outlined in **5.3.3.3,** as well as a monthly performance check (see **5.3.2**).

5.2.3.7 Gold Seal

- Replace annually.

- Post-maintenance check: Successful tune (see **5.4**) followed by a successful blank as outlined in **5.3.3.3,** as well as a monthly performance check (see **5.3.2**).

5.2.3.8 Helium Tank

- Replace as needed to ensure a supply of helium.
- Post-maintenance check: Successful tune (see **5.4**).
- NOTE: Instruments on an air handling system need not be documented in the GC-MS Logbook, and are exempt from the post-maintenance check.

5.2.4 Non-routine Maintenance

5.2.4.1 When non-routine maintenance is performed, the instrument shall be out of service until the non-routine maintenance is evaluated by the GC-MS Coordinator or designee to determine the need for additional instrument checks prior to analyzing samples.

5.2.4.1.1 If maintenance is performed that may affect retention times, a monthly performance check (see **5.3.2**) shall be performed before the instrument is placed back in service. This includes, but is not limited to, column changes.

5.2.5 Shutdown

5.2.5.1 A successful tune (see **5.4**) shall be performed following any GC or MS shutdown.

5.2.5.2 The shutdown shall be noted in the Activity Log.

5.3 Standards and Controls

5.3.1 Naming and Saving of Instrument Files *(".D" folders and all files contained therein)*

5.3.1.1 All instrument files created during performance checks, and all blanks and data files associated with case samples shall be saved on the instrument computer hard drive according to the year/month in which it was collected.

5.3.1.2 All instrument files listed above shall be placed into a compressed (.zip) file. The compressed file shall be named with the instrument identifier, year, and month in which it was collected.

5.3.1.3 The compressed (.zip) file shall be archived monthly in the FA object repository ("Manage Files") associated with the GC-MS instrument on which it was collected.

5.3.2 Monthly Performance Check

5.3.2.1 Two multi-component standard solutions, made up of a variety of drugs commonly encountered in the laboratory shall be injected on a monthly basis when the instrument is in use to verify instrument performance. The solutions

shall be run during the first seven calendar days of each month. Any instrument on which the standard solutions are not run during the first seven days of the month shall be out of service until the standard solutions are successfully run.

5.3.2.2 Additional standard solutions may be run on a monthly basis to establish retention times. Any additional monthly standard solutions may also be used to verify instrument performance following any maintenance performed.

5.3.2.3 The retention time of each required component of the standard solutions shall be compared to previous runs. Any shift greater than 1.0 % that cannot be attributed to maintenance shall be documented in the GC-MS Log and the instrument evaluated by the GC-MS Coordinator or designee. The instrument shall be taken out of service and steps shall be taken, such as column maintenance or other preventative maintenance, until the shift is no longer greater than 1.0%.

5.3.2.4 The mass spectrum of each required component in the standard solution shall be substantially the same as a reference material spectrum (see **5.7.3**). Any appreciable differences shall be noted in the GC-MS Logbook and the instrument evaluated by the GC-MS Coordinator or designee.

5.3.2.5 The total ion chromatograms for each standard solution shall be visually inspected for resolution between the required components. Any deficiencies shall be documented in the GC-MS Logbook and the instrument shall be evaluated by the GC-MS Coordinator or designee.

5.3.2.6 The Forensic Scientist reviewing the monthly standard solution injections shall document the retention times for each standard solution in the GC-MS Retention Time log. The reviewing Forensic Scientist shall mark the activity log to indicate the successful run of each standard solution.

5.3.3 Blank injections

5.3.3.1 Prior to the injection of a sample, a blank solvent injection shall be made using the same method and split ratio as the sample.

5.3.3.2 The solvent shall be prepared by the individual Forensic Scientist at the time of sample preparation and be the same solvent from the same bottle used in the sample preparation.

5.3.3.3 The blank solvent injection shall be evaluated to ensure that the instrument and solvent are free of the following:

5.3.3.3.1 Any controlled substance.

5.3.3.3.2 Any substance being identified in the sample.

5.3.3.3.3 Any substance that may interfere with the identification of sample component(s).

NOTE: The presence of large amounts of common gas chromatography peaks (e.g., siloxanes) shall be noted in the GC-MS Logbook and reported to the GC-MS Coordinator or designee.

5.3.4 Syringe flush

5.3.4.1 The syringe shall be flushed at least 10 times with solvent between injections to ensure the sample integrity between injections and to ensure that no sample transfer is made between sample vials.

5.3.4.2 Methanol shall be used in the first wash vial.

5.3.4.3 Hexane or chloroform shall be used in the second wash vial.

5.4 Calibrations (Tune) – MSD

5.4.1 Calibration (tuning) shall, at minimum, be successfully completed on a weekly basis, as well as prior to the monthly performance check or after performed routine maintenance as described in **5.2.3**, utilizing the Standard Spectra Tune.

5.4.1.1 A Standard Spectra Tune shall be performed according to specifications listed in Appendix B using Perfluorotributylamine (PFTBA) as the tuning standard.

5.4.1.2 Compare the tune report to previous tunes and specifications as outlined in Appendix B. Notify the GC-MS Coordinator or designee of any major variations.

5.4.2 A System Verification shall be completed daily prior to instrument use on days when a Standard Spectra Tune is not performed.

5.4.2.1 Compare the system verification report to previous system verification reports. Notify the GC-MS Coordinator or designee of any major variations.

5.4.2.2 The system verification report shall be considered successful if the report states “passes”.

5.4.2.3 The system verification report shall be considered unsuccessful if the report states “one or more specifications was out of range”. Notify the GC-MS Coordinator or designee. Steps shall be taken to address the parameters out of specification. A successful Standard Spectra Tune is required after a failed system verification report, and prior to the instrument being used for casework.

5.4.3 Neither a tune nor a system verification is required to be completed on days when instruments are not in use. Sample sequences that continue overnight may be allowed to complete without performing a new tune or system verification (as applicable) provided that they do not extend more than twenty-four hours beyond the time of the last tune or system verification, or noon, whichever is later.

5.4.4 Record each tune or system verification in the Activity Log along with initials, date, and any parameters that are out of specification.

5.4.5 Tune reports or system verification reports shall be maintained electronically.

5.5 Sampling – Refer to the Drug Chemistry Section Administrative Procedure for Drug Chemistry Analysis.

5.6 Instrument Procedure

5.6.1 If an instrument problem or error message occurs, the individual who discovers the problem shall notify the GC-MS Coordinator, or designee. If the problem cannot be immediately corrected, the Activity Log shall be marked to show that the instrument is out of service.

5.6.2 Sample Preparation

5.6.2.1 Refer to the Drug Chemistry Section Technical Procedure for Extractions and Separations.

5.6.2.2 Evaluate and prepare samples prior to injection to avoid overloading and the introduction of extreme pH, oil, sugar and compounds known to be retained in the instrument.

5.6.2.3 Solid samples shall be filtered with solvent to prevent particulate matter and undesired compounds from being introduced into the instrument (e.g., sugars). Particulate matter shall not be visible in an auto-sampler vial.

5.6.2.4 Derivatizing agents may be used when needed.

5.6.3 GC-MS Methods

5.6.3.1 When the standard methods are not appropriate to analyze a compound, a modified method may be used in accordance with the Laboratory Procedure for Authorizing Deviations.

5.6.3.1.1 In the event a new GC-MS method needs to be developed refer to the Laboratory Procedure for Validation of Technical Procedures and section **5.1.2** above

5.6.3.2 Descriptions of specific method parameters are located in Appendix C.

5.6.3.2.1 When GC-MS is being used as a screening technique, the GC method chosen shall screen for a wide variety of controlled substances, from phenethylamines to high molecular weight compounds such as JWH compounds and steroids.

5.6.3.3 Splitless injections are generally not utilized, but may be used for sample solutions that did not provide successful identification of a compound using a 5:1 or higher split ratio.

5.6.4 Sequences

5.6.4.1 The current date shall be used when naming a sequence. Sequences need not be archived.

5.6.5 Instrument Files

5.6.5.1 Instrument file names shall include the year designation and the case file number to ensure that files from different years with the same file number are distinguishable.

5.6.5.2 Instrument files associated with casework and performance checks shall not be deleted or overwritten.

5.6.5.3 Instrument files shall be archived as outlined in **5.3.1**.

5.7 Evaluation and Identification

5.7.1 The GC-MS provides retention time data and mass spectral data.

5.7.2 Evaluate the total ion chromatogram (TIC) for peaks of interest.

5.7.2.1 The data generated from an unknown substance shall be evaluated to ensure that it is suitable prior to comparison to known reference material or published spectral data.

5.7.2.2 Initial evaluation shall include the assessment of peaks in the TIC that have an abundance (peak height) of greater than 10,000 counts above the baseline.

5.7.2.2.1 Peaks fitting the criteria of **5.7.2.2** may be further evaluated for mass fragmentation patterns and ion distributions.

5.7.2.2.2 If the sample produces peaks not fitting the criteria of **5.7.2.2**, the mass fragmentation pattern may still be assessed to determine if additional steps need be taken to obtain peaks fitting **5.7.2.2**, including but not limited to changing the split ratio, concentrating the sample, or preparing a new sample.

5.7.2.3 The presence of major ions in the mass spectrum that are not present in all scans of the chromatographic peak may be indicative of background noise or co-eluting substances. Isolate the source of the additional ions and subtract prior to searching the reference collection of reference material mass spectra.

5.7.2.4 The sample mass spectrum shall be searched and compared to a reference collection of reference material mass spectra. Probability Based Matching (PBM) shall be used to aid the Forensic Scientist in the identification but shall not be used as the sole basis of identification

5.7.3 Mass Spectral Identification

5.7.3.1 The general distribution of ions and the relative abundances of ions observed in the reference standard shall be observed in the sample.

5.7.3.2 The mass spectrum must contain the major ions, to include the base peak, and diagnostic ions unique to the analyte.

5.7.3.2.1 All ions with a relative intensity greater than 10 % of the base peak in the reference standard spectrum must be present in the sample spectrum.

5.7.3.2.1.1 For compounds with no ions greater than 10% of the base peak, magnify the y-axis of the reference standard spectrum and the sample mass spectrum to aid the analyst in identifying the two next most abundant ions with a *m/z* greater than 100 in each spectra. Those two ions identified in the reference standard shall be present at a relative abundance consistent with the sample mass spectrum.

5.7.3.2.1.2 This magnified spectrum shall be included in the casefile.

5.7.3.2.2 When methamphetamine or phentermine are confirmed utilizing GC-MS in conjunction with preliminary testing, the retention time of a single sample shall be compared to the retention time of the respective reference material according to **5.7.4.3**.

5.7.4 Retention Time (RT) Identification

5.7.4.1 Retention time data shall be required for the following:

5.7.4.1.1 No preliminary tests are available for the substance identified by GC-MS.

5.7.4.1.2 Sample size does not allow for additional testing, other than GC-MS.

5.7.4.2 When sample size allows, a second sample shall be analyzed for mass spectral and retention time comparison purposes.

5.7.4.3 The requirement for retention time identification shall be retention time which, when compared to a reference material standard, has a difference of less than or equal to 0.05 minute. The retention time may be determined by using an integrator in the data analysis software or may be determined as the elution time at which the mass spectrum was collected.

5.7.4.4 The reference material standard shall be run within ninety days before or after the case sample. The interval between a sample and a standard injection shall not contain column maintenance.

5.7.5 For sample runs, the case record shall contain:

- Total Ion Chromatogram (TIC) for the corresponding blank.
- Total Ion Chromatogram (TIC) for the sample.
- Mass spectra of peaks of interest, such as common diluents, artifacts, or peaks comparable in size to any controlled substances present.
- Expanded mass spectra of phenethylamines and other compounds as needed.

- Mass spectra of any reference material standards used for identification and its unique identifier, and the corresponding library search.
- When retention time is required for identification of a compound, the blank and standard TIC as well as the mass spectrum of the reference material shall be included. Retention times shall be indicated for comparison purposes.

5.7.6 As an exception to **5.7.5,** sample runs not used due to blanks not meeting the requirements set forth in **5.3.3.3**, the case record shall contain at least the following:

- Total Ion Chromatogram (TIC) for the corresponding blank.
- Total Ion Chromatogram (TIC) for the sample.

5.8 **Reporting** - Refer to the Drug Chemistry Section Technical Procedure for Drug Chemistry Analysis.

5.9 **Calculations -** N/A

5.10 **Uncertainty of Measurement -** N/A

6.0 Limitations

6.1 The GC-MS methods described in this procedure cannot be used to distinguish between optical isomers or some positional isomers.

6.2 Introduction of improperly prepared samples may lead to poor sensitivity and carryover, and as such controlled substances should be confirmed within reason; sufficient abundance of the total ion chromatogram peaks needs to be achieved in order to produce acceptable spectra, without overloading the chromatographic system.

6.3 The 500LOW Methods cannot separate the co-elution of ethylone and alpha-Pyrrolidinopentiophenone (alpha-PVP), as well as some other co-eluting compounds. If these substances are encountered in combination in casework, other methods of isolation (such as derivatization) may need to be considered.

7.0 Safety

7.1 Refer to the Laboratory Safety Manual: Chemical Hygiene Plan and Hazardous Communication Program.

7.2 Handle syringes with care to avoid punctures.

7.3 Use extreme caution dismantling/installing/transporting compressed gas cylinders. Cylinders shall not be moved without the cylinder cap securely in place.

7.4 Gas Chromatograph and Mass Spectrometer may be extremely hot. Avoid touching hot areas and wear protective gloves while performing maintenance.

7.5 Refer to Appendix A for chemical hygiene and safety precautions for extremely hazardous and particularly hazardous substances.

8.0 References

Agilent GC Instrument Manuals (for each series used in the section)

Agilent MS Instrument Manuals (for each series used in the section)

Moffat, A.C., et al., eds. *Clarke's Isolation and Identification of Drugs*. 2nd Edition. London: Pharmaceutical Press, 1986.

Skoog, Douglas A., F. James Holler and Timothy A. Nieman. *Principles of Instrumental Analysis*. 5th *Edition*. Garcourt Brace & Company, 1998.

Agilent GC-MSD ChemStation and Instrument Operation Student Manual Course Number H4043A Volume 1, Revision E.02.xx. Agilent Technologies: printed February 2008.

Murray, K.K, R.K. Boyd, M.N. Eberlin, G.J. Langley, L. Li and Y. Naito. IUPAC Recommendations 2013. *Pure Appl. Chem.*, 2013, **85**, 1515-1609.

Laboratory Safety Manual: Chemical Hygiene Plan and Hazardous Communication Program.

9.0 Records

- GC-MS Log Book
- Case Record

10.0 Attachments

- **Appendix A** – Chemical Hygiene and Safety Precautions for Extremely Hazardous and Particularly Hazardous Substances
- **Appendix B**
- **Appendix C**

Revision History		
Effective Date	Version Number	Reason
04/19/2021	14	1.0 – Updated GC-MS model numbers 3.0 – Added definition for diagnostic ion 4.1, 4.2, 4.3 – Clarified GC-MS model numbers, added materials, updated ACS grade requirement 5.1.2.2 – Clarified criteria for performance verification 5.1.3 – Clarified GC-MS Activity Log and added requirement to record liner changes 5.2.2 – Added requirement for highlighted entry in Activity Log 5.2.3 – Updated septum change to monthly, added blank requirement after tunes, updated source cleaning schedule 5.2.4.1.1 – Added column change requirement 5.2.5.2 – Changed maintenance log to activity log 5.3.1.3 – Changed annually to monthly 5.3.2.3 – Changed 2.0% to 1.0% and added additional guidance 5.3.2.6 – Added GC-MS Retention Time Log and archival schedule 5.3.3.3 – Updated formatting 5.4 – Updated Tune schedule, removed references to Appendix B, added daily System Verification 5.6.1 – Reworded 5.6.3.2 – Removed reference to Appendix C 5.7.2, 5.7.3 – Added criteria for evaluation of data and clarified criteria for mass spectral identifications 5.7.4.3 - Reworded; changed RT difference from 0.1min to 0.05min 5.7.4.4 – Changed 30 days to 90 days 5.7.4 – Formerly 5.6.7; moved and expanded 6.1 – Added positional isomers 7.1-New 7.5-New 8.0-added Laboratory Safety Procedures 9.0 – Added GC-MS Retention Time Log New Appendix A Removed former Appendix B and D Former Appendix C became Appendix B – extended run times in LOW and 500LOW methods and retitled page

Appendix A – Chemical Hygiene and Safety Precautions for Extremely Hazardous and Particularly Hazardous Substances

Methylene Chloride/Dichloromethane **DANGER: PARTICULARLY HAZARDOUS SUBSTANCE** *	
	**HEALTH 2** **FLAMMABILITY 1** **REACTIVITY 1**
Detection of Release	Clear colorless liquid. Ether like odor
Signs/Symptoms of Exposure	Serious eye irritation; skin irritation; may cause drowsiness or dizziness.
PEL	ACGIH (TLV) – 50 ppm; OSHA Specifically Regulated Chemicals/Carcinogens – (PEL) 25 ppm
Associated Hazards	Serious eye and skin irritation; suspected of causing cancer
Controls	Use under fume hood. Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product. Use eye protection. Handle with gloves. Wear lab coat. Gloves: Fluorinated rubber (break through time = 148 minutes)
Safe handling, storage, disposal	Avoid contact with skin and eyes. Avoid inhalation of vapor or mist. Keep in a tightly closed container. Containers which are opened must be carefully resealed and kept upright to prevent leakage. Dispose of in Hazardous Chemical Waste.
Emergency Procedures (2.2)(4.1)(6)	**Eye Contact:** Rinse thoroughly with plenty of water for at least 15 minutes and consult a physician. **Inhalation Exposure:** If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician. **Ingestion:** Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician. **Skin Contact:** Wash off with soap and plenty of water. Consult a physician. **Spills:** Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Evacuate personnel to safe areas. Small contained spill: wearing appropriate PPE, collect with absorbent material, and place in container. Dispose in Hazardous Chemical Waste. Large spills: Evacuate area and call 911 (Haz Mat).

*__GHS Ratings (2.1)__:
Skin irritation (Category 2)
Eye irritation (Category 2A)
Carcinogenicity (Category 2)
Specific target organ toxicity - single exposure (Category 3), Central nervous system

__IARC:__
Group 2A: Probably carcinogenic to humans

Appendix B

Standard Spectrum Tune Parameters

1. The mass assignments of the three tuning masses shall be within +/- 0.2 amu of 69.00, 219.00, and 502.00. If the deviation is larger than +/- 0.2 amu, document the deviation on the tune and/or in the activity log. Perform another standard spectra tune. If the problem persists, document the deviation on the tune and/or in the activity log and notify the GC-MS Coordinator or designee. The instrument shall remain out of service until the problem is corrected.

2. The peak widths of the three tuning masses shall be 0.55 +/- 0.10 amu and the peaks shall generally be smooth and symmetrical. If the deviation is greater than 0.10 amu, document the deviation on the tune and/or in the activity log. Perform another standard spectra tune. If the problem persists, document the deviation on the tune and/or in the activity log and notify the GC-MS Coordinator or designee. The instrument shall remain out of service until the problem is corrected.

3. The base peak shall be identified as mass 69. The relative abundance ratio of mass 219 to mass 69 shall be within 40 – 85 % and the relative abundance ratio of mass 502 to mass 69 shall be within 2.0 – 5 %. If these requirements are not met, document the deviation on the tune and/or in the activity log. Perform another standard spectra tune. If the problem persists, document the deviation on the tune and/or in the activity log and notify the GC-MS Coordinator or designee. The instrument shall remain out of service until the problem is corrected.

4. The 70/69 isotopic ratio shall be from 0.5 – 1.6, the 220/219 ratio shall be from 3.2 – 5.4, and the 503/502 the ratio shall be from 7.9 – 12.3. If these requirements are not met, document the deviation on the tune and/or in the activity log. Perform another standard spectra tune. If the problem persists, document the deviation on the tune and/or in the activity log and notify the GC-MS Coordinator or designee. The instrument shall remain out of service until the problem is corrected.

5. The abundance of any peaks less than 69 amu shall not be greater than 10 % of the abundance of the base peak.

6. Peaks at 18, 28 or 32 amu are indicative of water, nitrogen and oxygen, respectively, and may indicate an air leak.

7. If an air leak is detected, the air leak shall be isolated and corrected and the tune repeated. Record the tunes and maintenance activity in the instrument logbook. If the problem persists, document the deviation on the tune and/or in the activity log and notify the GC-MS Coordinator or designee. The instrument shall remain out of service until the problem is corrected.

Appendix C

GC Method Parameters

Column dimensions: 30 m X 0.25 mm X 0.25 µm

HIGH METHODS – These methods are used for compounds that elute after 16min. in the screen method, such as some steroids and some synthetic cannabinoids. Method run time is 25 min. and the sample injection is 1µL. Scan range is 40-500 amu. The following are the specific methods used:

HIGH100 – 100 split, 1.00 minute initial time, 280 ºC initial temperature, 10 ºC/minute ramp, 300 ºC final temperature, 22.00 minute final time, 25.00 minute total run time

HIGH20 – 20 split, 1.00 minute initial time, 280 ºC initial temperature, 10 ºC/minute ramp, 300 ºC final temperature, 22.00 minute final time, 25.00 minute total run time

HIGH5 – 5 split, 1.00 minute initial time, 280 ºC initial temperature, 10 ºC/minute ramp, 300 ºC final temperature, 22.00 minute final time, 25.00 minute total run time

HIGHSL – No split, 1.00 minute initial time, 280 ºC initial temperature, 10 ºC/minute ramp, 300 ºC final temperature, 22.00 minute final time, 25.00 minute total run time

LOW METHODS – These methods are used for typical drug samples (cocaine/amphetamines/most opiates). It is used for compounds that elute before 16min. in the screen method. Method run time is 16min. and the sample injection is 1µL. Scan range is 40-500 amu. The following are the specific methods used:

LOW100 - 100 split, 1.50 minute initial time, 100 ºC initial temperature, 30 ºC/minute ramp, 300 ºC final temperature, 7.83 minute final time, 16.00 minute total run time

LOW20 – 20 split, 1.50 minute initial time, 100 ºC initial temperature, 30 ºC/minute ramp, 300 ºC final temperature, 7.83 minute final time, 16.00 minute total run time

LOW5 – 5 split, 1.50 minute initial time, 100 ºC initial temperature, 30 ºC/minute ramp, 300 ºC final temperature, 7.83 minute final time, 16.00 minute total run time

LOWSL – No split, 1.50 minute initial time, 100 ºC initial temperature, 30 ºC/minute ramp, 300 ºC final temperature, 7.83 minute final time, 16.00 minute total run time

SCREEN METHODS – These methods shall be used to screen samples when a substance is NOT previously indicated. Method run time is 35min. and the sample injection is 1µL. Scan range is 40-500 amu. The following are the specific methods used:

SCRN100 - 100 split, 1.50 minute initial time, 100 ºC initial temperature, 30 ºC/minute ramp, 300 ºC final temperature, 26.83 minute final time, 35.00 minute total run time

SCRN20 – 20 split, 1.50 minute initial time, 100 ºC initial temperature, 30 ºC/minute ramp, 300 ºC final temperature, 26.83 minute final time, 35.00 minute total run time

SCRN5 – 5 split, 1.50 minute initial time, 100 ºC initial temperature, 30 ºC/minute ramp, 300 ºC final temperature, 26.83 minute final time, 35.00 minute total run time

500LOW METHODS – (To be used on an as needed basis) These methods are used to improve resolution between structurally similar compounds, including but not limited to synthetic cannabinoids and

phenethylamines. Method run time is approximately 31 minutes and the sample injection is 1uL. Scan range is 40-500amu. The following are the specific methods used:

500LOW100 – 100 split, 2.00 minute initial time, 70 °C initial temperature, 15 °C / minute ramp, 275 °C final temperature, 15.00 minute final time, 30.67 minute total run time.

500LOW20 – 20 split, 2.00 minute initial time, 70 °C initial temperature, 15 °C / minute ramp, 275 °C final temperature, 15.00 minute final time, 30.67 minute total run time.

500LOW5 – 5 split, 2.00 minute initial time, 70 °C initial temperature, 15 °C / minute ramp, 275 °C final temperature, 15.00 minute final time, 30.67 minute total run time.

500LOWSL – No split, 2.00 minute initial time, 70 °C initial temperature, 15 °C / minute ramp, 275 °C final temperature, 15.00 minute final time, 30.67 minute total run time.