Title: Gas Chromatography/Mass Spectrometry (GC/MS)

A. **Introduction**:

Gas Chromatography/Mass Spectrometry (GC/MS) can be used to provide qualitative identification of analytes and, when applicable, quantitative information. The GC/MS instrumentation provides both retention time and fragmentation pattern information for analytes. Comparison of data (samples versus positive controls) provides the basis for qualitative identification. Using GC/MS data analytes can be tentative identified using library spectra for comparison. Confirmatory identification can be achieved by comparing both chromatographic and mass spectral data with known reference materials. Although the GC/MS is a single instrument it is considered two (2) separate orthogonal analytical techniques.

B. **Responsibility**:

All analysts who are authorized to perform casework ; preferably within the Drug Chemistry discipline.

C. **Definitions/Acronyms**:

QA/QC: Quality Assurance/Quality Control
m/z: mass-to-charge ratio
GC/MS: Gas Chromatography/Mass Spectrometry
CHEP: Cyproheptadine
EtOAc: Ethyl Acetate
MeOH: Methanol
$CHCl_3$: Chloroform
Instrument Check Solution: a substance put into a GC/MS sample that demonstrates the instrument had an acceptable injection and is working as expected. This is generally CHEP and the solvent is usually methanol, but other compounds or solvents can be used, if needed.

D. **Equipment/Materials/Reagents**:

1. General laboratory glassware
2. Gas chromatograph with mass spectral detector (GC/MS)), Agilent (or equivalent)
3. Perfluorotributylamine (PFTBA, FC-43) (Agilent or equivalent)
4. Capillary Column: (Restek™ Rxi™-5ms, 30m, 0.25mm i.d., 0.25µm, or equivalent)
5. Chloroform (reagent grade or equivalent)
6. Cyproheptadine (CHEP) (reagent grade or equivalent)

7. CHEP/MeOH (0.001% ; 10ppm): ~10mg of CHEP in ~1000mL of methanol (Instrument check solution ; need not be quantitatively accurate)
8. CHEP/EtOAc, (0.001%, 10ppm): ~10mg of CHEP in ~1000mL of ethyl acetate (Instrument check solution ; need not be quantitatively accurate)
9. Ethyl Acetate (reagent grade or equivalent)
10. THC: Tetrahydrocannabinol
11. Methanol (reagent grade or equivalent)
12. Various glassware and materials (variable suppliers)
13. Daily Standard: Fentanyl, Oxycodone, Phencyclidine (PCP), Cocaine, delta-9-THC, Heroin – in a CHEP solution. This is a mixture of drugs made from reference standards which can represent samples found within casework. The composition may change based on the needs of the unit but, if changed, should be documented within the instrument QA/QC log books.
14. Combo Control: amphetamine and trazadone reference standards in a CHEP solution.

E. **Preparation of Performance Test Solution, Controls (Standards):**

When preparing the stock Daily Standard Mix record the volumes used and lot numbers on the Daily Standard Stock Solution Reagent Log sheet (See CS-7.3). The log sheet is maintained in a log book within the Unit.

1. Daily Standard Mix (aka: Performance Test Mix)

The concentrations of analytes within the Daily Standard Mix should be:

Analyte	Stock Solution Concentration	Volume of Stock Solution	Total (or Final) Volume of CHEP solvent	Final Concentration of Analyte in Mix
Cocaine	1 mg/mL	100 µL (0.10 mL)	10 mL	10 µg/mL (10 ppm)
Fentanyl	1 mg/mL	100 µL (0.10 mL)		10 µg/mL (10 ppm)
Heroin	1 mg/mL	300 µL (0.30 mL)		30 µg/mL (30 ppm)
Oxycodone	1 mg/mL	100 µL (0.10 mL)		10 µg/mL (10 ppm)
PCP	1 mg/mL	100 µL (0.10 mL)		10 µg/mL (10 ppm)
THC	1 mg/mL	75 µL (0.75 mL)		7.5 µg/mL (7.5 ppm)

a. The date the solution is made will include the lot number assigned to the appropriate Daily Standard Mix solution. For traceability purposes, this date/lot number should be on any aliquots of the stock solution that will be retained for future use. The date the Daily Standard Mix solution was placed in service/use will be documented on form CS-7.3.

b. Since this solution will be assessed each day of use no expiration date needs to be listed on the Daily Standard Mix's container. If it is determined that the stock solution is no longer acceptable (either due to contamination or compound breakdown) then the stock solution, and any aliquots thereof, will be properly disposed.

Note-01: The Daily Standard Mix is re-verified each time GC/MS results are considered acceptable. The solution's expiration date is one (1) year from the last date of verification.

2. Positive and Negative Controls

a. Reference Standards (aka. Positive Control Solutions)

Solutions made from either solids or pre-made solutions (usually purchased as reference standard materials) which contain specific analytes (or multiple analytes) and are prepared in an appropriate solvent(s). Concentrations of these analyte solutions should be less than 100 µg/mL (100 ppm) for each analyte and be appropriate for comparing to analytes within unknown samples. Documentation of how such control solutions were prepared will be recorded and filed, as needed.

b. Residue Positive Control Solutions

Solutions of specific analytes (or multiple analytes) can be prepared in an appropriate solvent. Concentrations of these analyte solutions should be less than 50 µg/mL (50ppm) for each analyte and be appropriate for comparing unknown sample analytes of residual concentrations. Documentation of how such controls were prepared will be recorded and filed, as needed.

c. Control Materials (Positive or Negative)

Known substances (preferably a commercial product from a known vendor or from a known source) that can be used for comparison purposes. Historically these materials have referred to plant materials, rock materials, powders, residues, and foods from known sources – all with appropriately-known sourcing information (i.e., where obtained).

Positive Control materials are usually those which either contain (or which have been added/spiked with) known analytes. Examples of such positive control materials include foods spiked with drugs. These materials (usually extracts) should be analyzed in a manner similar to, and contemporaneously with, unknown samples for comparison purposes.

Negative control materials can be matrix-type materials that are similar to unknown samples and which do not contain analytes of interest (i.e., drugs) or which have not been spiked. Such controls are usually extracted/analyzed in a manner similar to, and contemporaneously with, unknown samples.

F. **Procedure**:

1. Set Up of GC/MS and Evaluation:

   a. GC/MS Daily check:

   Prior to an instrument being used for casework on any given day an air/water check, tune, and daily standard solution evaluation are performed. A check list can be utilized for documentation and to demonstrate that an instrument is acceptable for use. The checklist should be filed in the QA/QC maintenance log book for each instrument. (See CS-7.2)

Note-02: If QA/QC instrument log books become digital then an equivalent method of storage can be on electronic devices.

Note-03: If the instrument is still running a sequence from the night [previous day], instrument evaluation can be done at the conclusion of the sequence and no interruption is necessary simply because the days have changed).

Note-04: The term 'daily' only refers to the day an instrument is used for casework or similar type of work (e.g., procedure validation).

i. Tuning of Mass Spectrometer:

(a) Generally autotunes are performed the day instruments are used for casework, but tuning more frequently is acceptable.

Note-05: Quick tunes have in the past been acceptable but are no longer preferred.

(b) Tune Parameter Review:

(i) Correct mass assignments (m/z 69, 219, and 502) +/- 0.2

(ii) Peak widths (acceptable range 0.45-0.65)

(iii) Relative ion abundances (69 at 100%, 219 > 20%, and 502 > 1%)

(iv) EM voltages are recorded to help track the condition of the source.

Note-06: Relative Ion Abundances within autotunes are instrument dependent. The above criteria was set to allow acceptability among all GC/MS instruments within the Unit. The data within each autotune printout should be compared to previous autotune data *for the same instrument* to help determine if significant changes have occurred and instrument attention is necessary.

ii. Air and Water Checks:

These are performed each day of use to verify that there are no leaks in the GC system. The ions corresponding to water (m/z 18), nitrogen (m/z 28), and carbon dioxide (m/z 44) are checked to verify that values are less than 10% of m/z 69 and to track any trend that may suggest there is a leak.

iii. Daily Standard Mix (Qualitative)

At a minimum this usually consists of Cocaine, fentanyl. heroin, oxycodone, PCP, and THC in a CHEP solution (other appropriate drugs may accompany, if necessary). This mix is analyzed each day to verify that there are no unexplainable shifts in expected analyte retention times and that mass spectra are consistent to what is expected. The printout of the daily standard is maintained in a file in the instrumentation room (or equivalent).

Note-07: The Daily Standard Mix analytes may be altered based on the requirements of the Unit.

Note-08: The Daily Standard Mix is re-verified each time GC/MS results are considered acceptable. The solution's expiration date is one (1) year from its last day of verification.

iv. Instrumentation Evaluation

In the event of a problem within one or more of the evaluation parameters (air/water check, tune, or daily standard), analyst(s) will work to determine the cause of the failure prior to analyzing casework. This may include the need for basic maintenance. If the problem is corrected the check sheet is marked as acceptable for use and any troubleshooting or solutions should be documented. If the problem still occurs and the instrument should not be used, it will be marked appropriately as not acceptable for use. Analysts will work with the Lead Examiner to determine the issue with the out-of-service instrument and try to correct the issue. An appropriate 'Out-of-Service' sign will be placed on the instrument to indicate that it will not be used for casework.

All appropriate printouts (e.g., air/water checks, tunes, and the documentation of any maintenance performed) are located in maintenance (QA/QC) log books (or equivalent) for each instrument. Printouts of daily standards are kept in a file within the instrumentation room.

The following parameters are reviewed and documented on the GC/MS Daily QA/QC Maintenance Log (CS7.2)

(a) Evaluation of GC/MS: Expected analytes within the Daily Standard Mix (i.e., Target Peaks) will be present. Other detected analytes (i.e., Non-Targeted peaks) should not be present. If non-targeted analytes are present then they must be explainable (e.g., 6-MAM peak due to the breakdown of heroin)

(b) Evaluation of GC/MS: The ratio of abundance of heroin to CHEP should not decrease less than 20% from the previous day's acceptable data.

(c) Evaluation of GC: The retention times of each of the Daily Standard Mix analytes will not deviate by +/- 5% when compared to respective previously acceptable performance standard solution chromatographic data.

(d) Evaluation of MS: The mass assignments from the mass spectra of the components within the Daily Standard Mix must compare favorably (i.e., similar in peak profile) to known spectra and to previously analyzed spectra.

b. Instrument Maintenance:

Analysts may perform routine maintenance if trained. No non-routine maintenance (e.g., change or clip column, venting instrument) should be performed on an instrument without notification to the Lead Examiner. All maintenance (routine or otherwise) will be documented within the instrument QA/QC maintenance logbook.

i. Septa: Analysts can determine if septa need to be changed based on the results of instrument evaluation (i.e., air/water, Daily Standard Mix (or other sample)).

ii. Liners: Analysts can determine if liners need to be changed based on the results of instrument Daily Standard Mix (or other sample) evaluation. Decreased sensitivity for heroin (or other analytes) and/or the presence of unexpected peaks may indicate the need to change a liner.

iii. Liner seals: Analysts can determine if liner seals need to be changed based on the results of instrument evaluation (i.e., Daily Standard Mix (or other sample)).

iv. Clipping column/Changing columns: Analysts can determine if column maintenance is necessary based on the results of instrument evaluation (i.e., Daily Standard Mix (or other sample)). Loss of sensitivity or the presence of unexpected peaks may indicate the need for column clipping or changing.

v. Switching/changing filaments: Loss of current or complete/partial signal loss indicates a possible need for a filament change.

vi. Cleaning the source: An increase in electron-multiplier (EM) voltage values may indicate that the source needs to be cleaned.

vii. Other Maintenance: as required due to instrument function and based on the evaluation of instrument performance results.

c. Instrument Checks: Most samples prepared for GC/MS injection contain CHEP (cyproheptadine) in methanol or in an appropriate solvent. Samples (blank, sample, or control/standard) are placed in CHEP and the CHEP acts as an internal performance check during each sample injection.

(i) Blanks:

Solutions with no unexpected analytes. These are mainly used to verify that there is no carryover in-between injections (e.g., case samples).

1. A blank should be one of the first samples analyzed in a set of samples. This ensures the system is free of possible contaminates.
2. Blanks will be analyzed between different sample types in the same case.
3. Blanks should be analyzed at a minimum of every five (5) injections if multiple like-items from a single case are analyzed.
4. Blanks will be analyzed in-between different cases.

(ii) Controls:

1. Negative Control:
   Prepared in the same manner and at the same time as samples and with no analytes other than CHEP.
2. Positive Control:
   Usually prepared in a similar solvent matrix as samples and contain known analytes (along with CHEP) which can be used to compare to samples.

(iii) Additional Instrument Checks:

The CHEP and positive controls are used to show that the instrument is working properly as determined by the expected retention time and mass spectral fragmentation patterns. The addition of CHEP to samples allows analysts to determine if valid injections were made successfully.

Note-09: The internal CHEP can be made in solvents other than methanol if analytes of interest are not soluble in methanol or if methanol causes artifacts to be formed (e.g., designer amphetamines or derivatizations).

2. Sample Preparation:

Samples for analysis can range from a variety of matrices (e.g., powder, rock, residue, plant material, pill, tablet). Samples limited in size or amount/type of material can be rinsed (or extracted) with solvent (or otherwise prepared). Some of these samples may be considered

residues based on the limited amount of material that is present. Such materials include, but are not limited to, empty syringes, other types of drug paraphernalia, plant materials, tablets, liquids, and food stuffs. (See SOP CS-4).

a. Sample log: samples which are to be run by GC/MS are listed on a GC/MS Sample Position Worksheet (see CS-7.1). This is a map of sample position on the auto sampler. It is acceptable for GC/MS vials to only be labeled with the vial position (this is considered properly labeled), since this map will allow the identity of the sample to be tracked. The sequence printout can also be used to track the sample vials as long as the printouts are checked and documented.

b. Solid Samples (e.g., Powder or Rock):

   i. Add approximately 1 mL of solvent (e.g., CHEP/MeOH or CHEP/EtOAc, as appropriate) to properly labeled containers (e.g., GC/MS vials or test tubes).

   ii. Add ~1 mg sample (e.g., using a new applicator stick or clean spatula) to the vial (or test tube), mix, and ensure sample adequately dissolves into solution.

   iii. Cap the vials (or test tubes) in preparation for analysis.

   iv. Prepare appropriate blanks and controls (containing CHEP) for the analysis and in a similar manner as sample preparation. The negative control solution containing CHEP can also be considered a reagent blank.

Note-10: Because of the potential for variable sample concentrations, dilutions may be necessary to adjust concentrations of sample solutions so adequate data can be produced. Controls will be analyzed in the same manner as samples whenever possible. Dilutions of controls may be necessary. Documentation of any changes (e.g., dilutions) should occur.

c. Residue samples (including samples extracted or otherwise prepared so that they are now in a residual form)

   i. To the dried residue (often previously prepared in a GC/MS vial) add 1 mL of appropriate solvent (e.g., CHEP in MeOH or CHEP in EtOAc, as appropriate) and cap.

   ii. Prepare appropriate blanks and controls for the analysis.

Note-11: Because of the variable nature of residue samples, analyst discretion will be employed in this process regarding dilutions or concentrating samples. Unusual or exceptional cases should be discussed with the Lead Examiner or Deputy Director to ensure method applicability.

d. Tablets: tablet samples can be analyzed direct or as a residue if an extraction method was used.

   i. If directly analyzing then prepare samples as a Powder/Rock sample listed above. If analyzing samples after an extraction was performed then analyze samples as residue samples listed above.

   ii. Prepare appropriate blanks and controls for the analysis.

   iii. Manufactured tablets with an identifiable imprint will be analyzed by searching for drug information using the logo and reputable literature, whether hardcopy or online. Information obtained will be documented in the case file along with references. Such evidence may additionally be analyzed by other techniques (e.g., FTIR, GC/MS) for confirmation purposes, if necessary. Evidentiary tablet analyses can be stopped after information is obtained through a logo search only if tablets are not suspected to be illicitly produced and upon approval from either the Lead Examiner or higher. Subsequent reports shall convey the appropriate limitations to a logo-only search.

3. GC/MS Analysis:

   a. Each GC/MS is checked and evaluated on each day of use and prior to the analysis of any samples. If an instrument is still analyzing a sequence from the night before then QA/QC evaluation can be done at the conclusion of the sequence. Once an instrument is marked as acceptable for use it can then be considered acceptable for the day's casework unless proven otherwise. (see CS-7.2)

   b. It is acceptable to have changes to the temperature programs, as needed, for specific cases as long as the appropriateness of the change is demonstrated by properly analyzing blanks and controls. Any variations beyond a simple temperature hold in the temperature method will be documented in the case file, either by a notation or a printout of the temperature method. It is a good practice to print hardcopies of methods along with each sequence and place into case files. Vial placements can be verified by using the sequence printouts prior to analyses.

   c. Samples prepared as above should be injected using the appropriate GC/MS method (e.g., 'Drugs.m' or 'COMBO.m').

      i. The 'Drugs.m' method is appropriate for most routine samples. This includes plant extractions, most tablets, rocks, and powder samples. This method is not appropriate for most steroids since those usually contain late-eluting substances. It is also not recommended for some designer methamphetamine and amphetamine-type analytes since those are low molecular weight substances and require a slower rate in GC-oven temperature ramping.

ii. The 'Combo.m' method is a combined method that encompasses both early and late eluting analytes. This method is generally used for powder, tablet, and rock samples that are negative by the 'Drugs.m' method. This method is a longer method that will often detect early eluting substances such as amphetamines and late eluting compounds such as trazadone. When using this method an appropriate 'Combo-type' positive control should be analyzed.

iii. Copies/printouts of methods are maintained with each instrument so that instrument parameters can be recalled for specific sample sequences at any time. If a new method is developed a copy of that method will be maintained similarly.

iv. When there is an indication of the type of substance present (such as through a tablet imprint) the standards books can be consulted to see which temperature program is appropriate for the compound of interest.

d. Autosampler Sequence: After entering the sequence of samples, blanks, and controls into the instrument software, the information can either be written on the "GC/MS Sample Position Worksheet" (CS-7.1) or printed out. These sheets act as maps of the auto sampler so sample can be cross-referenced with data printouts. They can be kept on a clip board next to each specific instrument. Periodically these are removed from the clipboards and filed appropriately.

i. Vial position, method program, sample identification and instrument operator are entered into the sequence. The exact order of controls, reagent blanks, and samples may vary with each sequence, but generally the order of samples to be analyzed is:

(a) Negative control

(b) Positive control

(c) Blank

(d) Samples (usually no more than five (5) like samples are to be analyzed without a blank being injected)

Blanks or negative controls must be analyzed (unless approved by Lead or higher):
1) In-between different cases, or
2) In-between differing sample matrixes within a single case, or
3) In-between like sample matrixes if packaged in separate evidence bags.

(e) Blank

(f) Additional positive control reference standards, as needed.

Note-12: The Daily Standard Mix is acceptable for use as a positive control.

(i) Positive control reference standards are analyzed for the comparison of retention times (RT) and mass spectra (known to unknown). Even though a known positive control may have been analyzed, published spectra should be used and accompany analyte printouts from samples (i.e., library searching spectra) when possible.

(ii) Positive control reference standard solutions should be analyzed within 24 hours of the original injection and should be appropriate to the method that was used. However, if nothing has changed with the instrument or method that would affect the results (e.g., clipping column or temperature changes) then data from controls greater than a 24-hour period are acceptable, within reason.

4. Evaluation/Interpretation of Data:

   a. The total ion chromatogram (TIC) and associated spectra for each sample are reviewed using appropriate GC/MS software. Each significant chromatographic peak should be reviewed to determine if it is an analyte of interest (i.e., a controlled substance).

Note-13: Significant Peaks: To justify the existence of a significant chromatographic peak the peak's baseline signal-to-noise ratio (SNR), based on height, should equal or exceed three (3).

   b. Preliminary identification of an analyte is achieved by either operator recognition of a particular spectral profile or by the instrumental library providing matching spectral information.

   c. An identification is considered "confirmed" when the sample analyte is compared to a positive control (e.g., an in-house validated standard) and the following three (3) criteria are met:

      i. Chromatography

         Retention Times: Analyte peak retention times (e.g., relative retention times (RRT)) are within +/- 5% between sample and positive control.

Note-14: Exceptions may be made based on the analyte in question. For example, a wider window of acceptance (e.g., +/- 10%) may be accepted for steroids or non-controlled substances that do not chromatograph well (e.g., stanozol and acetaminophen).

ii. Mass Spectrometry

(a) Mass spectra will compare favorably to mass spectra from positive controls. Comparison of mass spectral fragment pattern ion ratios should also compare favorably but may differ based on certain factors (e.g., large concentration differences between controls and samples). Spectra may also compare favorably to known library spectra, although library spectra may have been produced under different mass spectral parameters.

(b) Mass spectral reference libraries (e.g., Wiley, AAFS, SWGDRUG, internal) will be used during the evaluation process. The internal library is made of substances analyzed in-house and may not be in the published libraries. Entries within the internal in-house library should be compared to a published spectra from a reliable source prior to use in casework.

Note-15: In some situations an analyte may be identified in a report by 'mass spectral match only' when an in-house standard is not available but a published reference spectra is available and is used for confirmation. It will be made clear in the report that the identification was made by using only mass spectral data and not chromatographic data (i.e., only one instrumental technique). This will generally only be used for non-controlled substances where GC/MS spectra are consistent with published reference data.

Note-16: Non-controlled substances, whether mixed with controlled substances or not, do not have to be identified. However, if analytes are found within a sample which are indicative of a 'cutting agent' and their chromatographic peaks are greater than peaks from controlled substances (or other analytes of interest), then the 'cutting agent' (or similar) analyte may be identified if deemed important by the analyst and proper controls are used. Contact an FSE2 or higher if uncertain whether to identify an analyte or not.

5. Acceptance of Data:

a. To accept sample GC/MS data the following must be true:

i. The blank prior to the injection of the sample must not contain significant unexplainable peaks. However, due the nature of samples analyzed within the Unit (e.g., plant materials or oil based samples), certain non-controlled substances may remain on a GC column even after the method has completed and thereby elute after multiple injections have been performed. If retention times significantly differ (i.e., minutes) then column carryover is likely the issue (as opposed to injection carryover)

and data may be acceptable. Consultation of an FSE2 or higher should occur in these situations if considering acceptance of data.

(a) If the analyte being identified in the sample is present in the blank prior to its injection then the abundance of the peak in the blank (height or area) must be less than 5% of the abundance of the CHEP peak, or the sample's analyte peak should be greater than ten-times (10x) that of the analyte peak from the previous blank.

(b) When it is known that there is a carry-over issue with a sample then multiple blanks can be injected to minimize the carryover and only the blanks after a sample and just prior to the next sample need to be evaluated.

(c) Blanks injected after a sample injection should be free of any analytes of interest. Any analytes found in the blanks may indicate that the method's temperature program was not long enough to elute all the peaks from the GC column.

ii. Controls must be acceptable.

(a) Negative controls will be free of analytes of interest and no significant unexpected peaks should be present. Explainable peaks will be acceptable.

(b) Positive control analytes must have acceptable chromatographic and mass spectral data with the unknown sample analytes for an identification to occur.

(c) Unexpected results within positive or negative control data should be reported to the Lead Examiner or higher for evaluation.

b. Documentation: Appropriate instrumental printouts, including reagent blanks and controls, will be included in the case file such that an independent reviewer would be able to readily interpret analyst conclusions.

i. The TIC (Total Ion Chromatogram) will be retained in case files. Label the chromatographic peaks for all analytes being identified and for the CHEP peak. Indication whether the retention times are acceptable should occur (e.g., *RT OK or RT* √).

(a) Every significant peak will be evaluated. Indications that peaks have been searched will be done either with a check mark or other method.

(b) Analysts may choose to label non-reported/non-confirmed analytes. This can be done by labeling the peak and putting the label in parenthesis or by making some other notation indicating it the analyte peaks have not been confirmed.

ii. All identified analytes will have documented spectra and appropriate comparative reference spectra (e.g., from positive controls and/or spectral libraries) within the case files.

iii. In samples where no analytes are identified the analyst should include the TIC along with any appropriate extracted ion chromatograms (EICs). If there are significant peaks that cannot be identified, or attributed to common artifacts, then such spectra should be included in the case file.

iv. In cases where identifications cannot be made due to the lack of obtaining a positive control, indications can be made , if appropriate (e.g., based on mass spectral library data).

v. Positive controls should be analyzed contemporaneously when identifying analytes in evidentiary samples or when specifically looking for analytes in samples which result in negative results.

GC/MS Common Temperature Programs (may be changed, if needed):

Temperature program	Drugs			Combo		
Initial temp	90° C			60° C		
Initial Time	0.00 min			0.00 min		
Ramps	rate	temp	Final time	rate	temp	time
Rate/final	30.0	300	7.50	20	200	0.0
				30	300	20.0
Post temp	0°c			280°c		
Post time	0.00			0		
Run Time	~14.5 min			~32.3 min		
Front inlet						
Mode	Pulsed splitless			Pulsed splitless		
Initial temp	250°c			250°c		
Pressure	9.32psi			8.19 psi		
Pulse pressure	40.0psi			40.0psi		
Pulse time	0.50 min			0.50 min		
Split Ratio	n/a			n/a		
Split Flow	n/a			n/a		
Total flow	48.1mL/min			53.4 ml/min		
Gas type	Helium			Helium		
Injection volume	1 microliter			1 microliter		
Post injection washes	Solvent A -3 Solvent B - 3			Solvent A -3 Solvent B - 3		
Tune file	ATUNE			ATUNE		
Acquisition mode	Scan			SCAN		

Solvent delay	4.95 minutes			3.73 min		
	Low mass	High Mass	Threshold	Low mass	High Mass	Threshold
	44	550	500	44	550	500
	MS Quad 150°c max 200°c			MS Quad 150°c max 200°c		

G. **References**:

1. Clark's Isolation and Identification of Drugs in Pharmaceuticals, Body Fluids, and Post-Mortem Materials, The Pharmaceutical Society of Great Britain.
2. The Drug Identification Bible
3. Baselt, R.C., *Disposition of Toxic Drugs and Chemicals in Man*, 7th ed., Biomedical Publications: Foster City, California, 2004.
4. Moffat, A.C., *Isolation and Identification of Drugs*, 2nd ed., Pharmaceutical Press: London, 1986.

Rev. #	History
3	Made slight grammatical and format modifications. Added a 'Revised History' section to the document. Updated logo searching part of the procedure. Strike-outs removed and color uniformly set as black. Replaced 'section' with 'unit.' Replaced section supervisor with Lead Examiner. Summarized steps so that they were not wordy. Replaced Laboratory with Unit. Corrected CHEP error from 1% to 0.001% concentration. Removed the option that the Daily Standard Mix solution's concentrations can be altered based on analyst's decision. Added that no maintenance should be done on instruments without Lead Examiner notification.
4	Added title to document. Changed Purpose to Introduction. General formatting changes throughout document. Removed 'Unit' within responsibility section. Updated definitions section. Updated 'Reagents' section to include other materials and instruments. Added 'Preparation of Performance Test Solution, Controls, and Standards' section and described how Performance Mix is prepared. Updated various parts of the 'Procedure' section to align with current practice and to improve clarity. Defined what a significant peak was and updated evaluation/decision criteria to reflect current practices. Updated tune parameter and methods. Updated 'References' section.