A. **Introduction**:

This procedure describes how to maintain, perform, evaluate, and verify instrument performance for acceptable analyses within the Controlled Substances Unit. In addition to the instrument performance criteria, this protocol provides guidance to the sample preparation and case work procedure for the analysis of evidentiary material within the unit. It is a combination of work instructions and general protocol. Gas chromatography (GC) and mass spectrometry (MS) instruments must be maintained in such a way as to verify their reproducibility and their reliability. One of the purposes of this procedure is to provide general guidelines for maintenance of gas chromatographs and mass spectrometers. Another purpose is to verify and track reproducibility, quality, accuracy, and reliability of routine instrument operation and data that are generated. Information is available to track instrument performance patterns for troubleshooting purposes.

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 tentatively identified using library spectra for comparison. Confirmatory identification can be achieved by comparing both chromatographic and mass spectral data with known reference materials.

The Agilent systems consist of a gas chromatograph with a single quadrupole mass selective detector (MSD) mass spectrometer, often referred to as a GC/MS. The mass spectrometer has a dedicated electron ionization (EI) source and may be referred to simply as an MS or MSD.

All analysts who use the GC/MS instruments within the Controlled Substances Unit will be responsible for following this procedure. If problems occur with instruments and they can't be resolved in-house then it is the responsibility of analysts to notify the FSE 2 (or higher) for alternative solutions (e.g., contact service engineer).

B. **Definitions/Acronyms**:

QA/QC: Quality Assurance/Quality Control
m/z: mass-to-charge ratio
GC/MS: Gas Chromatography/Mass Spectrometry
TIC: Total Ion Chromatogram
CHEP: Cyproheptadine
EtOAc: Ethyl Acetate
MeOH: Methanol
$CHCl_3$: Chloroform

C. **Equipment/Materials/Reagents**:

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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 Rtx-5MS, 30m, 0.25mm ID, 0.25μm, or equivalent)

5. Chloroform (reagent grade or equivalent)

6. Certified Reference Materials from Cayman Chemical, Cerilliant, Lipomed or another approved vendor (see solution preparation guidelines)

7. Ethyl Acetate (reagent grade or equivalent)

8. Methanol (reagent grade or equivalent)

D. **Preparation of Standard Solutions:**

Documentation of how solutions were prepared will be recorded and filed, as needed. Refer to CS-3 SOP for expiration date and stability information.

1. CHEP in MeOH (0.001%; 0.001mg/mL)

   a. Prepared by dissolving 10 mg of CHEP in 1000 mL of methanol.

2. CHEP in EtOAC (0.001%, 0.001mg/mL)

   a. Prepared by dissolving 10 mg of CHEP in 1000 mL of ethyl acetate.

3. CHEP in CHCl$_3$ (0.001%, 0.001mg/mL)

   a. Prepared by dissolving 10 mg of CHEP in 1000 mL of chloroform.

Note: volumes may be adjusted as needed.

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

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

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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		10 μg/mL
Fentanyl	1 mg/mL	100 μL		10 μg/mL
Heroin	1 mg/mL	300 μL	10 mL	30 μg/mL
Oxycodone	1 mg/mL	100 μL		10 μg/mL
PCP	1 mg/mL	100 μL		10 μg/mL
Delta 9-THC	1 mg/mL	75 μL		7.5 μg/mL

    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.

    b. Preparation information and the date that the Daily Standard Mix solution was placed in service/use will be documented on appropriate form.

    5. Combo Mix

Analyte	Stock Solution Concentration	Volume of Stock Solution	Total (or Final) Volume of CHEP solvent	Final Concentration of Analyte in Mix
Amphetamine	1 mg/mL	200 μL	10mL	20 μg/mL
Trazodone	1 mg/mL	1000 μL		100 μg/mL

    6. Reference Standards

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.

E. Performance Monitoring

Performance monitoring is essential not only to ensure the production of quality results but to avoid unnecessary sample loss and to minimize time wasted due to instrument inoperability. The daily standard test mixture is used to assess operating performance and continued integrity of the entire GC/MS system.

    1. Evaluate the tank pressure of the helium supply (carrier gas). The tank pressure gauge must read 100 psi or above. Change the tank prior to analysis if the tank contains 100 psi or less. The regulator should be adjusted so that the gauge for the pressure to the instrument reads ~60 psi.

    2. Perform an air/water check of the instrument. Evaluate the results using the 'Decision Criteria' section of this procedure. If the results are acceptable, print the air/water file and save in

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applicable instrument binder. If air/water check results are not acceptable perform necessary troubleshooting and maintenance and repeat check. If results are still unacceptable, consult an FSE2 (or higher).

    a.   Does not need to be performed if an autotune is needed. Follow step 3.

3.   If maintenance was performed on the mass spec or the instrument was vented for any reason: Perform a tune of the instrument. Autotune (ATUNE or ETUNE) should be used and the mass spectrometer will optimize its parameters automatically using PFTBA. Evaluate the results using the 'Decision Criteria' section within this procedure. If the results are acceptable then save and print the tune file (ATUNE or ETUNE) when completed. A second autotune may be necessary if the first tune produced questionable results or was unacceptable. If the tune results are still unacceptable after repeating a tune and corrections are unsuccessful then the instrument should be removed from service until corrected and an FSE2 (or higher) is notified.

4.   Run and evaluate the Daily Standard mixture using Drugs_SIM method. Evaluate the results using the 'Decision Criteria' section of this protocol. If the results are acceptable then print the TIC and store the printouts within the QC file.

5.   If the results are not acceptable then a second sample can be analyzed and evaluated. If the results are still unacceptable, and cannot be improved upon, then the GC/MS will be removed from service and tagged with a sign indicating 'Out of Service' until the problem is corrected. The FSE2 (or higher) will be notified of the issue.

6.   After the problem has been corrected the instrument in question must be checked and shown to pass evaluation before being returned to service.

H. **Decision Criteria**

    1.   Air and Water Check

| Air/Water Check: | $N_2 < 10\%$, $O_2 < 10\%$, $H_2O < 20\%$ |

    2.   Tune (ATUNE or ETUNE):

Evaluate MS performance using the criteria below. The results can be compared to tune results from previous tunes to indicate trends in instrument performance. Significant voltage increases or changes in the isotope ratios may indicate the need to initiate corrective maintenance procedures. The following are typical TUNE values for the MSD:

PFTBA Tune:	Mass +/- 0.2 for m/z 69, 219, 502
Peak Width (Pw50):	0.45 – 0.65
Relative Abundance:	69 = 100%, 219 > 40%, 502 > 2%
Air/Water Check:	$N_2 < 10\%$, $O_2 < 10\%$, $H_2O < 20\%$

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3. Daily Standard Test Mixture:

   a. In order for the instrument to be considered in good operating condition, all components of the daily standard mixture shall be present and generate Gaussian-shaped peaks. Other detected analytes must be explainable (e.g., 6-MAM peak due to the breakdown of heroin)

   b. The ratio of abundance of CHEP to Heroin constant when compared to previous run.

      i. Constant with is defined as a drop of not more than 20%

   c. The retention times of the components of the daily standard mixture will not deviate by +/- 0.1min when compared to the previous analysis of the daily standard mixture.

   d. The mass assignments from the mass spectra of the components of the daily standard mixture must compare favorably (i.e. similar peak profile) to known spectra and to previously analyzed spectra

   e. Heroin Spectral library match must be ≥90%

I. GC/MS Maintenance:

The following procedures are guidelines for performing GC/MS maintenance. All maintenance performed on the instruments shall be recorded within the appropriate binder or file.

1. GC Injector

   Regular replacements of the septum and liner will aid in reduction and removal of these undesirable interferences. The intervals for checking and replacement of septa and liners are 'as needed' and occur based on the data from the Performance Check Solution during QA/QC of the instruments.

   a. Septum and Liner Replacement

      i.   Set the oven to 30 °C.

      ii. Cool the inlet to room temperature and turn off the inlet pressure.

      iii. Remove the septum and liner retainer nut. Remove the old septum with tweezers and replace it with a new septum.

      iv. Remove the old liner from the injector with tweezers.

      v.   Place an o-ring on the new liner about 5-7mm from its top end.

      vi. Place the liner straight down into the inlet and replace the septum and liner retainer nut.

      vii. Restore the instrument conditions.

   b. Replacing the Inlet Gold Seal

      i. Cool the oven to room temperature and then turn off the oven.

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    ii. Cool the inlet to room temperature and turn off the inlet pressure.

    iii. Remove the column from the inlet.

    iv. Use a wrench to loosen the reducing nut and remove it. Remove the washer and seal inside the reducing nut.

    v. Replace the inlet gold seal and washer in the reducing nut.

    vi. Replace the reducing nut and tighten using a wrench.

    vii. Reinstall the column.

2. GC Corrective Maintenance.

   a. Clipping the column

    i. Set inlet and oven to room temperature.

    ii. Remove the column from the inlet and remove the column nut from the column.

    iii. Place the column nut and a new ferrule on the injector end of the column.

    iv. Score the column using a column cutter at six (6) or more inches. The score must be square to ensure a clean break.

    v. Break off the column end and inspect to ensure there are no burrs or jagged edges.

    vi. Position the column so it extends the required length (3-5mm) above the end of the ferrule.

    vii. Insert the column into the inlet and slide the nut and ferrule up the column to the inlet base. Finger tighten the column nut.

    viii. Tighten the column nut an additional ¼ to ½ turn so that the column cannot be pulled out from the fitting.

   b. Replacing the column

    i. Set inlet and oven to room temperature. Set the MSD to vent using instrument program.

    ii. After instrument has completed the vent cycle, power down GC then MSD. Remove column.

    iii. Place a capillary column nut and ferrule on the injector end of the replacement column.

    iv. Score the column using a column cutter. The score must be square to ensure a clean break.

    v. Break off the column end and inspect to ensure there are no burrs or jagged edges.

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    vi.   Position the replacement column so it extends the required length (3-5mm) above the end of the ferrule.

    vii.   Insert the column into the inlet and slide the nut and ferrule up the column to the inlet base. Finger tighten the column nut.

    viii.   Tighten the column nut an additional ¼ to ½ turn so that the column cannot be pulled out from the fitting.

    ix.   Install the column into the transfer line leading to the mass spectrometer. Column should be minimally visible from end of transfer line on MSD side.

3. MS Preventative and Corrective Maintenance

Lint-free gloves and appropriate personal protective equipment (PPE) must be worn during the disassembly and reassembly of the mass spectrometer. Appropriate PPE must be worn during pump oil changes. Source performance within the MS can be monitored by use of the performance monitoring solution. Although the preventative maintenance intervals are left to the analyst, minimal time frames are described.

a. Source Cleaning

The source shall be cleaned on MS systems as needed and based on system performance. The GC/MS systems require the removal of the entire source for cleaning to occur.

    i.   Vent the MS system and turn off the main power.

    ii.   Allow the source to cool before continuing.

    iii.   Open the vacuum chamber.

    iv.   Disconnect any appropriate lines or electrical connections to the source.

    v.   Loosen and/or remove source retaining bolts and clips and remove the source.

    vi.   Disassemble the source in order to separate the lenses and any surfaces that come in contact with the ionization chamber.

    vii.   Mix a slurry of aluminum oxide and methanol or deionized water.

    viii.   Thoroughly clean the pieces of the source with the slurry. Using a cotton-tipped applicator clean all dark or discolored areas, particularly around holes.
Warning: Only clean metal surfaces.

    ix.   Place the parts in a beaker containing deionized water and sonicate for at least (1) one minute.

    x.   Place parts in a beaker with methanol and sonicate for at least (1) one minute.

    xi.   Additional sonication steps using acetone followed by hexane may be performed as needed.

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xii. Re-assemble the source. Remember to avoid contaminating source/parts with oils from hands.

xiii. Place the source in the vacuum chamber and secure.

xiv. Reconnect all lines and electrical connections.

xv. Seal the vacuum chamber.

xvi. Turn on the main power and pump-down the system. Observe for vacuum leaks.

b. Filament Replacement

Generally a broken filament results in a total loss of ions rather than degraded system performance. To replace a filament:

i. Vent the MS system and turn off the main power.

ii. Allow the source to cool before continuing.

iii. Open the vacuum chamber.

iv. Disconnect any appropriate lines or electrical connections to the source.

v. Loosen and/or remove source retaining bolts and clips.

vi. Remove the source.

vii. Remove the old filament and replace it with a new one.

viii. Place the source in the vacuum chamber and secure.

ix. Reconnect all lines and electrical connections.

x. Seal the vacuum chamber.

xi. Turn on the main power and pump down the system, observing for vacuum leaks.

J. Quality Control:

All reagents, reference standards/products, and solutions thereof will be verified prior to use in casework analysis. Note: verification will consist of a successful analysis displaying expected instrumental data (e.g., chromatogram(s), mass spectral data, etc.) and exhibiting no unacceptable extraneous compounds. Appropriate positive and negative controls will be used with each sample or batch of samples. Appropriate reference standards/products will be used for casework comparison and/or controlled substance identification.

a. Negative Control or Positive Control

i. See CS- 4 SOP for guidance

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b. Reference Standards/Products

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

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.

c. Blanks

A blank is used to demonstrate that there is no carry-over contamination in-between samples during the instrumental analysis. Typically, a blank is simply a clean representative aliquot of the extraction/elution solvent.

i. A blank should be one of the first samples analyzed in a set of samples. This ensures the system is free of possible contaminates.
ii. Blanks will be analyzed between different sample types in the same case.
iii. Blanks should be analyzed at a minimum of every five (5) injections if multiple like-items from a single case are analyzed.
iv. Blanks will be analyzed in-between different cases.each sample, with the exception of two portions from the same sample.

K. Sample Preparation:

Samples for analysis can range from a variety of matrices (e.g., powder, rock, residue, plant material, liquid, tablet).   These procedures are used in conjunction with CS 4.1 and CS 4.2.

Note: Dilutions may be necessary. Documentation of any such changes should occur.

Sample log: The sequence printout is used to track the sample vials. The printouts are checked and documented.

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

i. Weigh ~1 mg sample and add to a test tube. Add ~1 mL of appropriate solvent, vortex, and ensure sample adequately dissolves into solution.

ii. Because of variable sample concentrations, using discretion, adjust concentrations of sample solutions by adding a portion of the sample solution to the appropriate CHEP solution (i.e. MeOH/CHEP, EtOAc/CHEP) in a GC/MS vial.

iii. Cap the vials and analyze.

b. Tablets:

If the tablet appears manufactured in nature with an identifiable imprint, search reputable literature (hardcopy or online) for drug information using the logo. Information obtained

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will be documented in the case file along with references and can be used as guidance for sample preparation.

    i.   Prepare samples as a Powder/Rock sample listed above.

        a) If information obtained in search indicates a low concentration sample, it is recommended to perform a borate buffer extraction using an appropriate solvent (EtOAc or $CHCl_3$).

    ii. Such evidence may additionally be analyzed by other techniques (e.g., FTIR, GC/MS) for confirmation purposes, if necessary.

  c. <u>Residue samples</u>

Samples limited in size or amount/type of material , including, but are not limited to, empty syringes, other types of drug paraphernalia, plant material, tablets, liquids, and food materials, including samples extracted or otherwise prepared so that they are now in a residual form. Evidence can be rinsed (or extracted) with solvent (or otherwise prepared),

    i.   Rinse the dried residue with ~1 mL of MeOH.

    ii. Because of variable sample concentrations, using discretion, adjust concentrations of sample solutions by adding a portion of the sample solution to the appropriate CHEP solution (i.e. MeOH/CHEP, EtOAc/CHEP) in a GC/MS vial.

    iii. The analyst may use discretion to directly rinse the residue evidence with MeOH/CHEP or EtOAc/CHEP if they have concern for the sample size for analysis.

  d. <u>Liquid Samples</u>
    i.   Aqueous liquids
        a) The analyst will select the sampling volume and solvent depending on the nature of the sample and any information available.
    ii. Oil-Based liquids
        a) The analyst will take into account the concentration and type of drug when selecting the sampling volume.
        b) Perform MeOH/Hexane extraction.

  e. <u>Plant Material Samples</u>
    i.   Plant material is soaked in ~0.5 ml of 1:1 Petroleum Ether/Methanol for a minimum of 30 seconds (no maximum time)
    ii. Approximately 1-2 drops of the resulting solution is drawn off and added to ~1mL of MeOH/CHEP solution in a labeled GC/MS vial for analysis.

  f. <u>Food Products</u>
    i.   Sugar based products: Recommend performing a Borate Buffer extraction
    ii. Oil based products: Recommend performing a MeOH/Hexane extraction.

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      iii. Amount of sample is dependent upon the suspected drug and any concentration information available.

  g. <u>Borate buffer extraction</u>
      i. Used to clean some manufactured tablets, for sugar based products, and to concentrate weak substances
      ii. Tablets, capsules, and powder/rock-like substances run and found to be negative may be run using a borate buffer extraction at the analyst's discretion.
      iii. Approximately 20-50 mg (maybe more depending on the nature of the sample) is placed in a test tube.
      iv. Approximately 1 mL of saturated Borate Buffer is added this is allowed to sit for a moment and then an approximately equal amount of extraction solvent is added (chloroform or ethyl ether are generally used).
      v. The solvent layer is transferred into a labeled test tube using a glass Pasteur pipette, a portion of which is added to the appropriate CHEP solution (i.e. MeOH/CHEP, EtOAc/CHEP) in a GC/MS vial. (The amount volume of the CHEP solution will depend on the nature of the sample).

  h. <u>Methanol /Hexane Extraction:</u>
      i. used to clean samples containing oils
      ii. Place 3-5 drops of the liquid or approximately 0.5 grams of sample (if food product) into a labeled test tube. Add approximately 2 mL of hexane-saturated methanol
      iii. Vortex 4-5 seconds.
      iv. Allow layers to separate. Carefully remove the lower, methanol layer using a glass transfer pipette and transfer this material to an appropriate CHEP solution (i.e. MeOH/CHEP, EtOAc/CHEP) in a GC/MS vial. (The amount volume of the CHEP solution will depend on the nature of the sample).

2. <u>GC/MS Analysis:</u>

  a. Hardcopies of methods along with each sequence will be placed into case files. Documentation of changes must be included with data so that any instrumental parameter change can be associated with data and casework until this procedure has been updated. Vial placements can be verified by using the sequence printouts prior to analyses.

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

      i. The 'Drugs_SIM' method is appropriate for most routine samples. This includes plant extractions, most tablets, rocks, and powder samples. This method was created as a general screen method with the additional SIM acquisition to monitor for m/z 245 (the base peak of fentanyl). The method is not appropriate for most steroids since those usually contain late-eluting substances nor for some designer methamphetamine and

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amphetamine-type analytes since those are low molecular weight substances and require a slower rate in GC-oven temperature ramping.

ii. The 'Combo' 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_SIM' method. This method is a longer method that will often detect early eluting substances such as amphetamines and late eluting compounds such as trazodone. When using this method, the Combo Mix will 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 reference standard data can be consulted to see which temperature program is appropriate for the compound of interest.

c. Autosampler Sequence: After entering the sequence of samples, blanks, and controls into the instrument software, the sequence is printed out and filed appropriately.

i. Vial position, method program, sample identification and instrument operator are entered into the sequence. The exact order of controls, 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

(e) Blank

3. Evaluation/Interpretation of Data:

a. The 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: 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.

Note: For identification purposes, there is no library quality match requirement. The instrumental library is used for guidance only.

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

i. Chromatography

(a) Retention Times: Analyte peak retention times (e.g., relative retention times (RRT)) are within +/-0.1 min between sample and reference standard.

Note: Exceptions may be made based on the analyte in question, consult with a FSE2 or higher.

ii. Mass Spectrometry

(a) Mass spectra will compare favorably to mass spectra from reference standards. 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) will be used during the evaluation process.

(c) Ion Ratios:

Refer to Section Mass Spectral Procedure for guidance on ion ratio acceptability criteria. Note: 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.

4. 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

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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 carryover issue with a sample, 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 will be retained in case files. The analyst has the option to label the chromatographic peaks for CHEP and all analytes being identified or to use the instrument software to print the Library Search Report.

(a) If labeling the TIC, 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.

(b) If using the Library Search Report, the analysts should document any peaks being identified that are not integrated by the software.

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.

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

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

References:

5. Clark's Isolation and Identification of Drugs in Pharmaceuticals, Body Fluids, and Post-Mortem Materials, The Pharmaceutical Society of Great Britain.

6. The Drug Identification Bible

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