Seized Drugs Technical Manual

Las Vegas Metropolitan Police Department
Forensic Laboratory
5605 W. Badura Ave. Ste. 120B
Las Vegas, NV 89118

Seized Drugs
Technical Manual
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| 10.1    | Abbreviations Key                        |
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**NOTE:** Hyperlinks were accurate at the time of manual publication.
“Controlled Substances” and “Seized Drugs” are synonymous titles of this Unit as they appear on forms, in manuals, as unique identifiers, etc.

Forensic Scientists assigned to the Seized Drugs Unit will be required to complete the training program that is defined in the Seized Drugs Training Manual. All Forensic Scientists, no matter their past work experience, must successfully complete a competency test and a mock court prior to supervised casework.

The primary objective of the Forensic Scientist in the Seized Drug Unit is to analyze physical evidence for the presence or absence of controlled substances. Controlled substances are those substances which are so designated either by Federal or State legislation or by administrative rules of the Nevada State Board of Pharmacy. The Uniform Controlled Substances Act is contained in the Nevada Administrative Code Chapter 453. It contains definitions, as well as an organized list of compounds that are enumerated into five schedules. The controlled status of a substance identified during analysis will be based on the booking date, not the analysis date.

The information in this manual was collected from numerous sources, including input from the Forensic Scientists and Managers of this laboratory system. In that regard, it meets the goal of providing the Forensic Laboratory with a workable guideline encompassing established facts, principles, and theories widely accepted by the general scientific community. The intent for the identification of seized drug samples within the Forensic Laboratory is to respond to the needs of the investigative agencies, the courts, and ultimately, the citizens they serve.

It should be noted that the nature of the sample(s) determine the analytical route the Forensic Scientist pursues and that many samples do not lend themselves to an exact order of analysis. Submitted evidence samples often contain excipients and diluents, therefore, analytical techniques are routinely modified or altered to characterize the sample successfully. The individual skills, judgment, and experience of the Forensic Scientists, coupled with the form, composition, and sample size of the exhibit determine which specific tests may be appropriate.

The analytical techniques presented in this Manual are offered as recommended procedures currently available and utilized for the analysis of suspected seized drug samples within the Chemistry Detail of the Las Vegas Metropolitan Police Department (LVMPD) Forensic Laboratory. The concept of a preset analytical scheme does not apply to the complexity of seized drugs analysis.
The analytical techniques currently available for use in the Seized Drugs Unit are:

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The Forensic Laboratory facility provides adequate environmental conditions to conduct all tests listed in this Technical Manual.

The Forensic Scientist must take adequate precautions to eliminate the possibility that the positive identification of a controlled substance could be due to inadvertent contamination in the laboratory. Every safeguard must be taken to ensure that samples are not contaminated with any extraneous material. The integrity of the sample(s) shall not be compromised.
1.1 Title: COLOR TESTS

Chemical color tests are nonspecific screening tests that provide presumptive data as to the nature of the substance. Typically these are performed at the beginning of an analysis. If the size of the sample is sufficient, perform the appropriate color test(s) required to provide an indication of any compound(s) that may be present.

PROCEDURE:
The color tests are conducted by transferring a small amount of a substance to a well on a clean porcelain or disposable plastic spot plate or into another appropriate vessel (e.g. test tube, filter paper, etc.). A drop of the chosen color reagent(s) is then placed into the well. A description of any color or lack of color which is observed shall be documented in the case file contemporaneously at the time of testing.

The case notes shall clearly indicate how many samples were color tested per item. The case notes can reflect either the number of samples tested or which specific samples were tested. For example: If Marquis was performed on nine (9) of the twelve (12) samples, the result can be written as “Result: [color] x 9” or as “Result: [color] (1)-(9).” If only one unit in the item is being tested, it is not necessary to indicate the number of units tested. Documenting the color result is sufficient and it is implied that only one (1) sample was tested.

If the same color test is repeated on a sample, an additional color test entry must be added to the case notes. This additional color test indicates that the same color test was repeated on this particular item a second time. For example, the Marquis color test was performed two (2) different times on the same item, which consists of six (6) packages. The case notes will reflect:

<table>
<thead>
<tr>
<th>Test:</th>
<th>Result:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Marquis [C072016-1]</td>
<td>Orange x 6 or Orange (1)-(6)</td>
</tr>
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</table>

Color test(s) information and/or results will never be deleted from case notes. If the color test is repeated any number of times, then every additional test(s) must be documented in the case notes. The color test results will not be altered without an explanatory note added to the case notes.
ADVANTAGES AND LIMITATIONS:
There is always a certain amount of subjectivity that must be taken into account when a color is reported. It is acceptable for different analysts to describe the same color differently. Aside from the differences in reporting colors that can be attributed to the analyst, colors can also be influenced by the concentration of the sample, the presence of contaminants, or the age of the reagent. The length of time during which the colors are observed may also influence the color reported because color transitions and instabilities are not unusual. Allowances should, therefore, be made for these differences.
1.2 Title: MICROCRYSTAL TESTS

Microcrystal testing generally involves the reaction of an analyte with a reagent, followed by microscopic examination of the morphology (shape) of the resulting crystals.

PROCEDURE:
Microcrystal tests can be performed by placing a minute amount of the sample on a microscope slide (in some cases the sample must be dissolved in a solution such as dilute acid) and adding a drop of the reagent to it. A second technique is the “hanging drop” method where the unknown is placed in a well of a microscope slide or a spot plate and a drop of the reagent is (placed on a cover slip) suspended over the solution. The solvent used in the well of the microscope slide is chosen so that the sample can be volatilized. In both methods, the resulting precipitates (crystals) are then observed using a compound microscope or polarizing light microscope at a suitable magnification. The most appropriate microscope and magnification will be determined by the analyst. A description or illustration of the resulting crystal formation shall be documented contemporaneously at the time of testing.

All microcrystal test reagents are for one time use and will be quality control checked after each preparation, either before or concurrently with use. The quality control check will include performing the selected microcrystal test on a known reference material that is believed to be the same as the unknown sample. All quality control checks will be documented in the case file.

The case notes shall clearly indicate how many microcrystal test samples were performed or the specific samples that were tested. For example: If the phosphate crystal test was performed on nine (9) of the twelve (12) samples, the result would be written as “Result: [shape and color] x 9” or as Result: [shape and color] (1)-(9).”

Microcrystal test(s) information and/or results will never be deleted from case notes. If the microcrystal test is repeated any number of times, then every additional test(s) must be documented in the case notes; the microcrystal test results will not be altered without an explanatory note added to the case notes.

ADVANTAGES AND LIMITATIONS:
Microcrystal tests are generally very sensitive, require very small sample amounts and in some cases are highly specific. The tests can also be a practical means for determining a specific enantiomer of an enantiomeric pair. Crystal tests are limited because the assessment of crystal morphology is subjective, and some crystal tests are not very specific for certain classes (i.e., opium alkaloids). Excipient material often distorts crystal formation and structure.
1.3 Title: CHROMATOGRAPHY

Chromatography is an analytical tool used to separate mixtures into individual components, and requires that a sample be dissolved in an appropriate mobile phase. Compounds are separated based on differences in their interaction between a stationary phase (solid or liquid coating on columns or plates) and a mobile phase (gas or liquid). The chromatography techniques used in the Seized Drugs Unit include thin-layer chromatography (TLC) and gas chromatography (GC).

THIN LAYER CHROMATOGRAPHY (TLC):
In thin-layer chromatography (TLC) the sample is spotted onto a TLC plate (a solid support [e.g., glass, aluminum, plastic, etc.] coated with an adsorbent) and then developed in a tank containing an appropriate mobile phase (solvent system) to effect a separation of components. Retention data for TLC may be determined for each individual system by running known reference materials on the same plate. Individual components are visualized by observing the plate under UV light or by spraying with a chemical that reacts with the individual components to produce a colored spot. TLC plates are properly disposed of after each use and TLC solvent systems are replenished as necessary.

General TLC Procedure:
1. Extract samples appropriately based on the analyte(s) of interest.
2. Spot a TLC plate with the appropriate sample(s), blank(s), and reference material(s) on the line of origin using glass capillary tubes.
   - Ensure the reference material has been retested within seven (7) days of running the TLC plate.
   - Spots should be in a straight line across the plate, approximately 1-1.5 cm from the bottom, and spaced far enough apart to prevent overlap (~6 mm apart). It is helpful to mark out where the spots will go using a ruler and a pencil and to label each spot.
   - There must be a blank for each extraction used on the samples being run on the same plate.
3. Add the appropriate solvent system to a TLC developing chamber that will fit the size of plate being used.
   - The level of the solvent system must be below the line of origin on the plate.
4. Add the plate to the developing chamber and place the lid on the chamber.
   - The plate should be placed so that the bottom is as close to the vertical edge of the chamber as possible while still being able to lean the top of the plate against the edge of the chamber.
5. Allow the plate to develop until the solvent system approaches the top of the plate. Avoid letting the solvent reach the top ~1 cm of the plate.
6. Remove the plate and allow it to dry.
   - Ultraviolet light can be used to determine if the plate is dry.
7. Visualize the spots on the plate appropriately.
8. Compare the staining and the relative distance traveled by the sample(s) and reference material(s) spots, and record the results in the case record.
   - If a sample and a reference material generally migrate the same distance and stain in a similar manner, results shall be considered consistent.
   - If a sample and a reference material do not migrate the same distance or stain in a similar manner, the result shall be considered inconsistent.

**TLC System Information:**

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Reference Material(s)</th>
<th>Solvent System</th>
<th>Visualization</th>
</tr>
</thead>
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<tr>
<td>THC</td>
<td>∆9-THC</td>
<td>4:1 Petroleum Ether:Diethyl Ether</td>
<td>Fast Blue 2B Spray</td>
</tr>
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</table>

**Methods:**
New procedures must be validated before use. New equipment, instruments, or methods that do not require a validation must be performance checked within the Chemistry Detail before use. Validated and performance checked method documentation will be stored electronically, in the Chemistry Laboratory and/or Qualtrax.

**Advantages and Limitations:**
TLC is a rapid and inexpensive screening method which can be used as a comparative technique. TLC requires little sample preparation and uses minimal amounts of sample. The technique can also be used as a method of separating compounds for further analysis (preparatory TLC). No specific structural identification of any compound can be obtained from TLC.

**Data Interpretation and Acceptance Criteria:**
The staining and the relative distance traveled by the sample and reference material will be evaluated. The color of the stains should be comparable, taking into account concentration differences. The two (2) stains must travel generally the same distance, taking into consideration that other compounds present in the sample extract can cause anomalies in the stain pattern.

**GAS CHROMATOGRAPHY**
In gas chromatography, the sample is dissolved in an appropriate solvent and injected onto the GC column. The GC column generally separates the sample into its individual components. Retention data for gas chromatography may be used as a screening test. Reference materials used for comparison must be analyzed on the same instrument and method as the unknown. The data generated shall reflect the data acquired, and will be stored in the appropriate case record within the LIMS.
Calibration and Maintenance:
A general column test check solution, generally consisting of an approximated concentration of at least three (3) reference materials in an appropriate solvent, will be run on the GC instrument on the first day of the week that it is being utilized, prior to any casework samples being run. The data from this run will be stored on each instrument’s computer.

When a new lot of the general column test check solution is prepared, a quality control check must be performed on a GC/MS instrument to verify that each component can be conclusively identified. This quality control check data will be saved in the LIMS Resource Manager. The prepared solution shall be stored in a chemical resistant glass container in the freezer. Reference materials that are expired per the manufacturer will not be used to prepare any column test check.

For methods associated with headspace analysis, a known chemical will be run on the headspace method on the same day as the casework sample(s). This known chemical will be considered the column test check for this method, and the data shall be included in the case file(s). The general column test check solution does not need to be run if the headspace method is the only method used that week on the instrument. The known chemical will be stored according to the manufacturer’s recommendations.

GC data for the column test check, including peak shape and retention times, will be evaluated to ensure that the instrument is performing adequately.

Routine maintenance will be conducted as needed and documented in the appropriate instrument log book.

Methods:
New procedures must be validated before use. New equipment, instruments, or methods that do not require a validation must be performance checked within the Chemistry Detail before use. Validated and performance checked method documentation will be stored electronically, in the Chemistry Laboratory and/or Qualtrax.

Advantages and Limitations:
GC requires little sample preparation and minimal sample quantities. GC cannot be used for components which are thermally labile or cannot be volatilized, however, derivatization can be used to improve the chromatography of such compounds, and can also give additional data for characterization and identification.

Data Interpretation and Acceptance Criteria:
If the chromatography shows symmetrical peak shape, the difference between the GC retention times of the sample and reference material will not exceed ±0.10 minute. If the chromatography shows an asymmetrical peak shape, then the GC retention time used for comparison may be outside the ±0.10 minute window, as long as the correlating mass spectrum meets the criteria listed in the Mass Spectrometry Chapter.
1.4 Title: MASS SPECTROMETRY (MS)

Mass spectrometry is a category A instrumental technique that provides structural data to aid in the identification of unknowns. Upon introducing a sample into the mass spectrometer (MS), the sample is ionized and fragmented by an accelerated beam of electrons or chemical radicals. The ions are then filtered according to their mass to charge (m/z) ratio by a scanning quadrupole magnetic field. A detector and data system (computer) record the mass and quantity of ions as the spectrometer is scanning, generating a mass spectrum.

PROCEDURE:
A sample may be introduced into the mass spectrometer by coupling it with a GC equipped with a capillary column. The sample is dissolved in an appropriate solvent and injected onto the GC column. The GC column separates the sample into its individual components which then enter the ion source of the MS. The computer is used to acquire data in a form which can be used by the analyst. This data will be stored in the appropriate case record within the LIMS.

CALIBRATION AND MAINTENANCE:
The mass spectrometer is tuned by an instrumental (computer) program which adjusts the MS operating parameters to optimize sensitivity and the mass/charge scale, monitor detector performance, and provide diagnostic information for troubleshooting. This tuning typically utilizes perfluorotributylamine (PFTBA), a stable compound which produces ion fragments throughout the mass range for the spectrometer. These ions are used to normalize the instrument’s operation. The instrument will be tuned on the first day of the week that it is being utilized, prior to any casework samples being run.

An electronic copy of the tune data will be kept on the instrument’s computer. Maintenance performed is documented in the instrument’s log book.

Previous tunes may be viewed as a chart so trends can be detected. The analyst may view this chart for all GC-MS instruments, except CS #3, by clicking on “CHECKOUT” from the main instrument view screen, then selecting “View Previous Tunes” from the top menu bar. For CS #3 GC-MS, click on “QUALIFY” from the main instrument view screen, then select “View Tunes” from the top menu bar.

Routine maintenance will be conducted as needed and documented in the appropriate instrument log book.

METHODS:
New procedures must be validated before use. New equipment, instruments, or methods that do not require a validation must be performance checked within the Chemistry Detail before use. Validated and performance checked method documentation will be stored electronically, in the Chemistry Laboratory and/or Qualtrax.
DATA INTERPRETATION AND ACCEPTANCE CRITERIA:
GC-MS data used for the identification of unknowns will be evaluated for acceptability by analyzing the overall peak shape and resolution, and by comparing the mass spectrum of the sample unknown to a reference spectrum. In general, peaks should be roughly symmetrical, however, it is understood that some compounds and matrices may preclude such criteria; the analyst should be able to articulate why their data still meets quality criteria.

Chromatography peaks should be of sufficient abundance to produce an acceptable mass spectrum. The sample spectrum will be compared to a known reference spectrum, using the following criteria:

- Ions across the spectra shall exhibit general correspondence of abundances and nominal m/z values.
- The spectra should have the same base peak unless variations based on abundance, carrier gas, etc. have been deemed acceptable by the laboratory.
- The spectra shall have the same molecular ion, when observed. There are times when certain compounds may not produce an observable molecular ion under EI mass spectral conditions. In such cases, comparisons can still be conducted based on the fragment ions present in the spectra.
- The presence of extraneous ions in the sample spectrum shall be evaluated to determine their significance.
- The absence of ions in the sample spectrum shall be evaluated for acceptability.
  - The absence of low abundance ions may be an acceptable difference between the spectra.
  - The absence of an ion, whose presence is useful for comparison or identification, is not acceptable.
- The reference spectrum used for comparison must be included in the case file.
1.5 Title: INFRARED SPECTROSCOPY (IR)

Infrared spectroscopy (IR) is a category A technique that provides structural data to aid in the identification of unknowns. It is based on the absorbance/transmittance/reflectance of infrared light energy corresponding to the vibrational and rotational transitions within the molecules of a sample. Infrared light containing wavelengths from 4000 cm⁻¹ to 400 cm⁻¹ is generated and passed through the sample. When the frequency of light matches a frequency of vibration within the molecule, absorption occurs. The absorptions are translated electronically and recorded on a data system.

PROCEDURE:
Samples may be examined using attenuated total reflectance (ATR), KBr pellets, or salt plates. Vapor phase samples are examined using a gas cell accessory containing a KBr window. The data will be stored in the appropriate case record within the LIMS.

METHODS:
New procedures must be validated before use. New equipment, instruments, or methods that do not require a validation must be performance checked within the Chemistry Detail before use. Validated and performance checked method documentation will be stored electronically, in the Chemistry Laboratory and/or Qualtrax.

CALIBRATION AND MAINTENANCE:
Verification that the FTIR is working properly will be done on a daily basis when the instrument is being utilized, prior to any casework samples being analyzed. The verification will be performed by running the qualification software (e.g. ValPro) supplied with the instrument. The data will be stored electronically on the instrument’s computer. Routine maintenance as recommended by the operator’s manual will be conducted on a regular basis and documented in the instrument log book.

PERFORMANCE CHECK PROCEDURE
A performance check on the instrument will be documented daily, when it is being utilized. Both the maximum and minimum values of the interferogram will be recorded on the quality assurance log sheet.

INFRARED SPECTROSCOPY PERFORMANCE CHECK PROCEDURE

A. Performance Check
   1. Load the OMNIC operating program
   2. Remove the diamond ATR attachment
   3. Replace with the FTIR cover
   4. Load Experiment: Transmission
5. Click OK
6. Select Collect on the menu bar
7. Select Experiment Setup
8. Click on the Bench tab, set the gain to 1 (change from Autogain to 1, do not save this change)
9. Click on the Diagnostic tab
10. Select Align
11. Record the Max and Min values in the FTIR Maintenance and Repair Manual after the alignment has been completed
12. Select OK

B. A validation software package, OMNIC ValPro, is included with the operating software for the Nicolet 380 FTIR. This program verifies whether the FTIR system is functioning properly or not.

1. Select Analyze on the menu bar
2. Select ValPro Qualification
3. Select Qualify
   This test takes approximately 10 minutes. The program runs several tests without the intervention of the operator.
4. Review the ValPro Qualification Report for pass/fail status
5. Close the report window
6. Save the report as a .pdf in the ValPro folder under the appropriate month and year

If the system fails any of the tests, close the window. Exit the program (OMNIC) and then reload the program again. Repeat the previous procedure (align the bench and revalidate the system). If the system continues to fail, seek further assistance.

Note: If using the Transmission/Transmission E.S.P. experiment, go to section C. If using the Smart Orbit ATR experiment, go to section D.

C. It is necessary to reload the experiment Transmission/Transmission E.S.P.:

1. Select Collect on the menu bar
2. Select Experiment Setup
3. Select Open
4. Select Transmission E.S.P.
5. Select open
6. Select OK

Proceed to section E.

D. Replace the FTIR cover with the diamond ATR attachment

1. The Smart Orbit experiment should load automatically
2. Click OK
3. The Smart Accessory test will run automatically
4. If the “all tests passed” message appears, click OK
5. Clean diamond cell and anvil. Do not use methanol

Proceed to section E.

E. Performance check

1. Prepare to collect a background and the polystyrene standard
   a. Click the ‘Col Smp’ graphical user interface button
   b. The first window titled ‘Collect Sample; Enter the Spectrum Title’ appears. Rename the file as “Polystyrene Test [date].” Click OK
   c. The second window titled ‘Information; A background spectrum must exist to provide the specified final format’ appears. Click OK
   d. The third window titled ‘Confirmation; Please prepare to collect the background spectrum’ appears. Click OK, then click ‘Start Collection.’ The instrument will now collect a background sample
   e. Once the background collection is complete, the fourth window titled ‘Sample; Please prepare to collect the sample spectrum’ appears. Place the 1.5 mm polystyrene standards into the pellet stand or onto the ATR base. Place the cover over the chamber or lower the diamond. Click OK, then click ‘Start Collection.’
   f. The fifth window titled ‘Confirmation; Data collection has stopped, add to window#?’ appears. Click Yes

2. Click the ‘Search’ graphical user interface button to compare the IR spectrum obtained to a library match

3. Go to ‘Report,’ then ‘Preview/Print Report’ and click ‘Print’
   a. Ensure that the date, time, instrument name and analyst’s P# and initials appear on the spectrum
   b. Save the file as a .pdf in the “Polystyrene” folder under the appropriate month and year
   c. On the ‘FTIR QA Log’ put a check under the ‘Polystyrene Tested’ column for that day
   d. Save the raw data file in the “Polystyrene” folder under the appropriate month and year

PROCEDURE FOR ADDING SPECTRA TO THE FTIR/ATR LIBRARY

All data must be approved by the Chemistry Manager/designee prior to entering it into the library.

1. Open the Omnic software on the computer for the FTIR/ATR instrument.
2. In the Omnic software, open the file you wish to include into the FTIR/ATR library.
   a. From the toolbar, select “File” then choose “Open…”
   b. Select the file.
   c. The spectrum will appear in the window.
3. From the toolbar, select “Analyze” then choose “Add to Library…”
4. A window titled “Select Library” will appear, choose “LVMPD C/S #6 ATR” and then click “OK.”
5. In the next window, at a minimum, enter the compound name, manufacturer and lot number.
6. Click “OK.”
7. Place the approved copy of the data in the reference material binder located next to the computer. Open the binder to the tab marked “C/S #6 ATR” and file the data alphabetically.
8. Make a copy of the data file.
   a. From the toolbar, select “File” and choose “Save As…”
   b. From the “Save in:” drop-down list, select “ATR Spectrum 2008”
   c. Rename the file to that of the reference material.
   d. If the name of the reference material already exists, add clarifying information such as the manufacturer, or the current date
   e. Click “Save.”

**NOTE:** Per Omnic software, “When you add a spectrum to a user library whose resolution is lower, the spectrum is deresolved. For this reason it may be better to save your sample spectra as files on the hard disk and consider the copies of them added to a user library to be for searching purposes only.”

**DATA INTERPRETATION AND ACCEPTANCE CRITERIA:**
A clean, clear, and full spectrum is essential for making comparisons.

A weak spectrum can be the result of poor sample preparation or interferences from other substances/matrix. In some instances, isolating the target analyte, or performing dilutions or concentrations may be necessary to improve response.

Once the case sample(s) has been run, the sample spectrum must be compared to a reference spectrum from a reputable source. The wavenumbers and relative intensities of the peaks present should generally agree between the reference and the sample spectra. Extra and/or missing peak(s) and peak anomalies should be evaluated.
X-Ray Fluorescence (XRF) spectrometry is a category A technique that can help to identify elements found in a substance or mixture.

**PROCEDURE:**
A sample will be placed in an approved XRF sample cup with plastic film attached, or may be placed directly onto the instrument. The data will be stored in the appropriate case record within the LIMS.

**METHODS:**
New procedures must be validated before use. New equipment, instruments, or methods that do not require a validation must be performance checked within the Chemistry Detail before use. Validated and performance checked method documentation will be stored electronically, in the Chemistry Laboratory and/or Qualtrax.

**CALIBRATION AND MAINTENANCE:**
The XRF instrument must be system checked prior to use each day. The manufacturer approved system check will be used and documented each time it is performed in the XRF spreadsheet located in LIMS.

**DATA INTERPRETATION AND ACCEPTANCE CRITERIA:**
Each element has a signature spectrum of energies (keV). The detected energies will be compared to the table of energies listed in the instrument software. The intensity (counts per second) of each peak will be evaluated by the analyst to determine if the elemental response is sufficient.

**PROTOCOL FOR THERMO NITON XRF:**

**Safety**
Put the thermoluminescent dosimeter ring on the finger that will be closest to the XRF window
The door of the XRF stand must be closed for the XRF instrument to operate

**Set Up**
Connect the XRF unit to the wall using the appropriate cord
Connect the XRF unit to the back of the base unit using the appropriate cord
Connect the XRF unit to the computer
Turn the XRF on by holding the power button for several seconds
Allow the XRF unit to warm up for approximately five (5) minutes
Launch NDTr software on the computer
Start Up

Press the screen of the XRF instrument to log in

![Warning]

**Caution Radiation**

THIS EQUIPMENT PRODUCES RADIATION WHEN LIGHTS ARE FLASHING.

Press YES to proceed.

Press NO to log out.

[Yes][No]

Press “Yes” when asked “Press YES to proceed”

![Enter Password]

Enter four (4) digit password (1234) and then press “Enter”

**Performing System Check:**
- Press icon “System Check” on screen
- Press “Yes” when prompted “Perform a System Check”

- Press “Close” when “System OK 100%” appears on screen

- Document in LIMS that the system check was completed

If the system check fails, see Corrective Action for the XRF instrument found under section 2.1 of this Technical Manual

**Performance Check:**

***Must be completed daily before use***

- Press “Sample Type” on screen
• Press “Soils & Minerals” on screen

• Press “Mining Cu/Zn” on screen

• Press “Data Entry” on screen
- Type applicable information into screen for Sample, Location, Inspector, Misc., and Note

- Press ↑ in the lower right corner of screen to return to previous menu

- Press “Tools” on screen

- Press “Element Range” on screen

- Verify that Mode is “Mining” and that all four (4) ranges (Main, Low, High and Light) are all checked and the Time must be listed as “30.0” for all ranges.

- Press “Save” on screen

- Place manufacturer prepared blank sample cup labeled “NIST 2709aPP 180-649” over the XRF window
- Either hold XRF instrument trigger for 120 seconds –OR– when attached to the computer press the button on the right side labeled “Batch”

***If using the computer, after selecting “Batch” make sure the number readings = 1 and duration = 120. Then select “OK.” The scan will start immediately.

If using the computer, press the “Esc” button after the spectrum has been collected and displayed on the screen. You may have to hit “Esc” multiple times to return to the proper screen. If using the XRF instrument press the lower right black triangular key (not on screen) with the back arrow embossed on it

- Press “Data Entry” on screen
- Type applicable information into screen for Sample, Location, Inspector, Misc., and Note
- Place manufacturer’s metal disc Copper Foil over the XRF window
- Either hold XRF instrument trigger for 120 seconds –OR– when attached to the computer press the button on the right side labeled “Batch”

***If using the computer, after selecting “Batch” make sure the number readings = 1 and duration = 120. Then select “OK.” The scan will start immediately.

If using the computer, press the “Esc” button after the spectrum has been collected and displayed on the screen. You may have to hit “Esc” multiple times to return to the proper screen. If using the XRF instrument press the lower right black triangular key (not on screen) with the back arrow embossed on it.

**Sample Collection:**
- At least one (1) blank must be run prior to running samples for each case

- Press “Data Entry” on screen
- Type applicable information into screen for Sample, Location, Inspector, Misc., and Note
- Place sample cup over the XRF window
- Either hold XRF instrument trigger for 120 seconds –OR– when attached to the computer press the button on the right side labeled “Batch”

If using the computer, press the “Esc” button after the spectrum has been collected and displayed on the screen. You may have to hit “Esc” multiple times to return to the proper screen. If using the XRF instrument press the lower right black triangular key (not on screen) with the back arrow embossed on it.
Transferring Data After Running All Performance Checks and Samples:

- Make sure that the XRF Analyzer is connected to a computer that has the Niton software.

- Turn on the XRF Analyzer.

Note: Wait approximately 30 seconds after turning on the XRF Analyzer to begin downloading files. The System Start screens do not allow downloading.

- Access the Niton Data Transfer (NDTr) software from Start menu → Thermo Scientific.

- Click the Download button. The Download dialog box will open.

A pop-up window informing that the connection tested successfully. If the test fails, there is a problem with the serial port setup. If the test fails, select the “Settings” button. A drop down menu will appear. Try selecting different ports from the drop down menu until the computer recognizes the XRF.

- In the Download dialog box, click the Connect button.
• Click the Query Readings button. This will return a list of all current readings on the analyzer. The list appears in the large white box in the Download dialog box.

• Select the readings to download. There are two ways to do this.
  o (1) Click the boxes next to each of the reading numbers to select or de-select individual readings. Select a range of readings by pressing the shift key, then selecting the first and last reading in the range. All readings from the first reading selected to the last will then be selected.
    - [ ] 1 ALLOY Reading
    - [ ] 2 ALLOY Reading
    - [ ] 3 ALLOY Reading
    - [ ] 4 ALLOY Reading
    
  o (2) You can also click the boxes on the left to select or de-select all the readings of a specific type.
The download generates a data file containing the selected readings. To save the file for later use:

- Enter the path for the file in the Destination Folder field. Use the ... button to browse.
- Enter a name for the file in the File Name field.
- Click the Download button.
- When the progress bar shows that all the readings are downloaded, click the Done button.

Any problems with downloading refer to instrument manual located H:\CB\Forensics\Chemistry\TRACE\Instrument Manuals

The data will not be deleted from the XRF analyzer until the raw data files are saved in the appropriate Unit Record Object Repository.
Deleting Data:

Quality control procedure for monitoring the XRF
Each time the Niton XRF is used, the NIST 2709a PP 180-649 sample cup and Copper foil Lot #B09S002 will be analyzed with the preceding operation procedures.

The data generated will then be added to the spreadsheet titled “Thermo XRF QC Tracking” located in LIMS Resource Manager inside the Manage Files folder for Niton XL3t GOLDD+. This Thermo XRF QC Tracking spreadsheet requires the elemental values for NIST 2709a PP 180-649 sample cup to be entered. A line graph for each element is generated as new data points are added, allowing the analysts to track and monitor trends. At the bottom of this spreadsheet is where the analyst will enter the keV and percentage values determined by the XRF when the Copper Foil is tested. An acceptable range has been determined and is displayed on the spreadsheet for both values. If the copper keV and/or percentage does not fall within the acceptable range(s) then the analyst will follow the corrective action listed in Chapter 5.0 – Quality Control Plan.
LVMPD FORENSIC LABORATORY
TECHNICAL PROCEDURES
SEIZED DRUGS

1.7 Title: RAMAN SPECTROSCOPY

Raman spectroscopy can be used as a category A technique when it is incorporated into laboratory grade equipment. The basic theory behind Raman technology involves passing a laser through a sample and measuring the resultant scattered wavelengths. The resultant wavelengths (called Raman scattering) are different than the incident wavelength, a shift which is caused by structural groups within the molecules in the sample. The pattern of scattering is detected and plotted on a spectrum for analysis and comparison to a known reference.

PROCEDURE:
The handheld Raman units and the benchtop Raman instrument allow samples to be scanned directly through clear or light colored packaging. The sample package can be lightly pressed against the nose cone of the handheld units or the microscope lens of the benchtop instrument for data collection. The benchtop instrument may require some focusing prior to data collection. Dark colored substances result in a high level of fluorescence and should not be analyzed using Raman spectroscopy.

METHODS:
New procedures must be validated before use. New equipment, instruments, or methods that do not require a validation must be performance checked within the Chemistry Detail before use. Validated and performance checked method documentation will be stored electronically, in the Chemistry Laboratory and/or Qualtrax.

CALIBRATION AND MAINTENANCE:
Verification that the benchtop Raman is working properly will be done on a daily basis, when the instrument is being utilized, by running the internal tests in the software supplied with the instrument. The data will be stored electronically on the instrument’s computer.

The handheld Raman units will be self checked prior to scanning each lab item number. The scan number of the self-check (for Tru-Narc only) and the scan number of the sample scans will be documented in the case notes.

Raman microscope maintenance as recommended by the operator’s manual will be conducted as needed and documented in the instrument log book.

DATA INTERPRETATION AND ACCEPTANCE CRITERIA:
A clean, clear, and full spectrum is essential for making comparisons.
A weak spectrum can be the result of poor sample preparation or interferences from other substances/the matrix. In some instances, isolating the target analyte, or performing dilutions or concentrations may be necessary to improve response.

Once the case sample(s) has been run, the sample spectrum must be compared to a reference spectrum from a reputable source. The relative intensities and the wavenumbers of the peaks present should generally agree between the reference material and the sample spectra. Extra and/or missing peak(s) and peak anomalies should be evaluated.

**BRUKER SENTERRA RAMAN MICROSCOPE DAILY VALIDATION PROCEDURE:**

**Power On:**

Turn on the controller box using switch in the back and push the “Standby” button.

Open the microscope door and turn on the switch on the right side. (Be careful to wait a few seconds after turning on the controller box. If performed too quickly, there can be communication errors with the software.)

Open the Opus software on the attached computer and input the password.

Click “OK” in the “About Opus” window that pops up.

**Daily Validation:**

In the ribbon, select “Validation” and then select “Run OVP Tests” from the drop down menu.

In the “Run OVP Tests” pop up window, select the laser and resolution from the IT drop down (e.g. “785 High Res”).

Check laser setting on the right side of the microscope marked “Laser Setting” to ensure that the laser selected from the drop down and the laser setting match.

Select the box marked “Raman PQ”.

Select “Run selected tests”.

A “Dialog” window will pop up and prompt you to set up slides to complete the daily validation.

Open the microscope door and place the slide onto the stage.

Ensure that the 50x objective is selected.

Switch to bright field (“BF”) on the front of the microscope and adjust the light control on the controller box as necessary.
View the sample through the optics and adjust the focus using either the stage controls or the joystick.

Close the microscope door.

Switch back to dark field ("DF") and click continue in the pop up window.

The software will perform the tests and prompt for any other slides required for the daily validation. Follow the same steps listed above to place and focus the slide on the stage and click “Continue”.

Once the daily validation is complete, print the test results and sign each page. Place the test into the logbook and complete the log sheet.

If all the tests pass, the instrument for the selected laser and resolution is ready for use.

If any of the tests fail, check the settings and ensure that each sample is properly focused and testing is performed in dark field setting. Repeat the testing. If the testing fails twice more after all corrections, call for service.
THERMO SCIENTIFIC TRUNARC ANALYZER PROCEDURE:

***This is considered a preliminary (Category B) test only***

Power on the TruNarc
- Press and hold the bottom key.
- When the ‘Activate Laser’ screen appears, enter 3-3-1-2 to unlock the instrument.
- The screen will flash green, ‘Laser Activated’ will appear, followed by the ‘Welcome’ screen.

Self-Check Process
- Ensure the nose cone is attached to the laser aperture.
- Move the attached self-check standard to the closed position.
- Select ‘Check’ from the ‘Welcome’ screen.
- Select ‘Check’ from the ‘Self Check’ screen to begin the self-check.
- A green background and ‘Pass’ appears when the device is working according to manufacturer’s specifications.
- See Recording Results section below for documenting results.
- Press the ‘OK’ button to return to the ‘Welcome’ screen and proceed to step 2.e.
- A red background and ‘Fail’ appears when the device is not working according to manufacturer’s specifications. Press the ‘OK’ button, and:
  - Verify that the Self-Check standard is securely in place.
  - Determine if the nose cone needs to be cleaned.
  - To clean the nose cone and Self Check standard, remove the nose cone from the device and rinse under clean running deionized water with the Self Check standard in the open position.
  - Dry the nose cone and replace it on the device.
  - Ensure laser aperture is not dirty/blocked.
  - Determine if the nose cone needs to be replaced.
  - Repeat Self Check Process
  - If the device continues to fail the self-check, mark the device out of service, document the issue in the device logbook located in Resource Manager, and contact the manufacturer.
  - Move the Self Check standard to the open position.

Scan Process
- Select ‘Scan’ from the ‘Welcome’ screen
- Position the sample
  - For samples in a plastic bag, press the bag to the edge of the nose cone.
  - For a loose sample, pile the sample and place the edge of the nose cone just above the sample, being careful not to touch the sample with the nose cone.
  - For samples in a thin-walled container (such as a clear glass vial), remove the nose cone and press the container against the laser aperture.
  - Do not scan dark colored samples, or through thick-walled or opaque/frosted containers.
  - Select ‘Scan’ from the ‘Scan Ready’ screen. The scan will begin immediately.
• The laser indicator light will turn off when the scan finishes, and the device software will begin to analyze the data.
• Document in the case notes the scan number (located in upper left corner of display screen) and the result.
• Select ‘OK’ from the ‘Scan Result’ screen.
• If more samples are to be scanned from the same lab item, repeat the scan process for each sample.
• If a different lab item will be scanned, perform the self-check process.

Review scans (if necessary)
• Select ‘Review’ from the ‘Welcome’ screen.
• Scroll through scans by pressing the up and down arrows. Select ‘Home’ from the ‘Review Scans’ screen to return to the ‘Welcome’ screen

Power Off the TruNarc
• Press and hold the power button for 10 seconds.

Recording Results
• The following information must be documented in the case notes when using this device:
  • Instrument’s unique identifier
  • Self-check scan number and result
  • Sample scan number and result

Storage and Transportation
• The TruNarc analyzer should be stored in the designated carrying case to protect the device when it is not in use and when being transported.

Data backup
• Electronic data is backed up on an external hard drive when the device’s data storage is full. This external hard drive is stored in the Drug Vault.
B&W TEK TACTICID ANALYZER PROCEDURE:

***This is considered a preliminary (Category B) test only***

**Power on the TacticID**
- a. Press and hold the Power On/Off key for approximately 2 - 3 seconds.
- b. When the “Enter Password” screen appears, enter 1-1-1-1 to unlock the instrument.

**Performance Validation Process**
- a. Go to Home Screen by pressing the Home Key.
- b. Replace the Port Protection Cap with the Validation Cap
- c. From the Home Screen, select “Setup”
- d. On the next screen, select “Perf. Validation”
- e. On the “Perf. Validation” screen select “Acquire”
  - i. A “Pass” appears when the device is working according to manufacturer’s specifications.
    - 1. See Recording Results section below for documenting results.
  - ii. A “Fail” appears when the device is not working according to manufacturer’s specifications. Press the “Back” button, and:
    - • Verify that the Performance Validation cap is securely in place.
    - • Ensure laser emission aperture is not dirty/blockaded.
  - iii. Repeat Performance Validation Process
  - iv. If the device continues to fail the performance validation, mark the device out of service, document the issue in the device logbook located in Resource Manager, and contact the manufacturer.

**Scan Process**
- a. Place the appropriate sampling accessory onto the device.
- b. Position the sample to optimize coverage of the chosen sampling accessory
  - i. Do not scan dark colored samples, or through thick-walled or opaque/frosted containers.
- c. From the Home Screen, select “Scan” from the Scan Ready screen, and press the Laser key. The scan will begin immediately.
- d. Document in the case notes the scan index (located in the center part of display screen) and the result.
- e. Press the Home Key to return to the Home Screen.
- f. If more samples are to be scanned from the same lab item, repeat the scan process for each sample.
g. If a different lab item will be scanned, perform the Performance Validation process.

Review scans (if necessary)
   a. Press the “View” key from the Home Screen.
   b. Performance Validations are not logged on the device, so they will not appear in the Scan History List

Power Off the TacticID
   Press and hold the Power On/Off key for approximately 2 -3 seconds.

Recording Results
   The following information must be documented in the case notes when using this device:
   • Instrument’s unique identifier
   • Performance Validation result
   • Sample scan index and result

Storage and Transportation
   The TacticID analyzer should be stored in the designated carrying case to protect the device when it is not in use and when being transported.

Data backup
   Electronic data is backed up onto a laptop which will be saved onto the LVMPD network for long term storage approximately every twelve (12) months.
REFERENCE MATERIALS:
All reference materials shall be verified prior to use by comparison to a reliable reference source including but not limited to:
- Forensic Drug Review
- AAFS library
- Wiley library
- Instrumental Data for Drug Analysis
- SWGDRUG library
- NIST library
- Another reference material of the same substance but different manufacturer or different lot# that has been previously verified

Neither the data provided on the certificate of analysis nor reference materials from the same manufacturer and lot number may be used for the verification. The verification will be documented in the LIMS Resource Manager and will include the manufacturer, lot number, the certificate of analysis, and MSDS/SDS when applicable.

Verified reference materials which are used to create a Forensic Laboratory library associated with each instrument shall include the manufacturer and lot number of the reference material in the library entry. Printouts of the GC-MS and FTIR reference materials library data will be kept in a binder at the instrument site or electronically.

All reference materials, with the exception of those used for quality control checks, must be retested prior to use. A category A technique must be used when retesting reference materials. The data generated must be compared to a NIST reference spectrum, internal library or another reliable reference source and will be uploaded into the object repository for that lot# in LIMS.

If a certified reference material (CRM) is changed in a way that alters the traceable measurement value (e.g. concentration), the equipment used (pipettes) to alter the CRM shall be traceable.

Reference material solutions prepared with chemicals that have since expired do not need to be disposed of because reference materials are retested prior to use.

Reference materials that are diluted are not considered a reagent preparation. Therefore, they do not require documentation in Resource Manager or in the case record, nor do they require assignment of an internal lot number. At a minimum, these diluted preparations will be labeled with the identity of the reference material(s), manufacturer, and lot number.
Safe Handling, Use, Transportation and Storage:
Reference materials will be handled and used with appropriate personal protective equipment and are not typically transported outside the Forensic Laboratory. Primary reference materials should be stored according to manufacturer’s recommendations inside the Chemistry Detail drug vault. Secondary reference materials should be stored in a cool, dry place in the Chemistry Detail drug vault.

If a reference material is transported outside the Forensic Laboratory, the container must be properly closed and labeled with the identity of the contents, lot number, proper Global Harmonization System (GHS) labels and any other pertinent information. During transport the reference material should be stored in the same manner as being stored in the Chemistry Detail drug vault.

A primary reference material vendor list is available electronically in Qualtrax.

SUPPLIES:
Approved vendors for purchasing of supplies will be evaluated. Vendors should be ISO Guide 34 accredited, however, if they are not, the following conditions may be considered to determine the vendor’s suitability:

- Experience with the vendor’s products and/or services as determined from past performance.
- Quality of products/services provided by the vendor
- Ability of the vendor to provide services/products in the necessary time frame

If a vendor is not ISO Guide 34 accredited, the Chemistry Laboratory Manager may create a memo attesting to the appropriateness of the vendor.

A record of the evaluation and/or a copy of the vendor’s ISO Guide 34 accreditation certificate will be maintained with the vendor list in Qualtrax.
LVMPD FORENSIC LABORATORY
TECHNICAL PROCEDURES
CONTROLLED SUBSTANCES

2.2 Title: REFERENCE MATERIAL INVENTORY AUDIT

Controlled substance reference material use will be documented in the Drug Weight History spreadsheet located in Qualtrax, using gross weights and/or tablet/capsule counts.

An annual inventory audit will be conducted on controlled substances and non-controlled substances. Any discrepancies in quantities or weights exceeding ±5% of the amount recorded in the Chemical Inventory/Drug Weight History will be brought to the attention of the Chemistry Laboratory Manager.

When performing the drug inventory audit, all fields in the electronic inventory and Resource Manager shall be verified for accuracy and consistency, including but not limited to:

- Spelling of product name(s)
- Lot number
- Location stored
- Container size
- Manufacturer name and information
- Accuracy of synonyms
- Quantity on hand
- Controlled status
- Type of reference material (primary or secondary)
- Inventory date

In addition to verifying all fields in the Chemistry Inventory, the following should also be reviewed and updated as necessary:

- Format of entries
- Gross weights of controlled substances
- Tablet/capsule counts of all pharmaceutical preparations
- Storage conditions meet manufacturer guidelines

PROCEDURE FOR CONTROLLED REFERENCE MATERIAL AUDIT

It is not necessary to complete each step in the order presented, but all steps must be completed.

An annual inventory audit will be conducted on all controlled reference materials. The information maintained in the Chemistry Inventory and the LIMS Resource Manager will be checked and updated. Gross weights will be measured and updated on the “Drug Weight History” page located in Qualtrax.
1. Ask the Chemistry Manager to archive the current version of the Excel Chemistry Inventory by saving a copy in the appropriate “Archive” folder that only Managers and above have access to.

2. In Qualtrax, check out the current version of the Drug Weight History spreadsheet by selecting the “Edit” option and listing a reason. Immediately check the document back in without making any changes. This will save a copy of the sheet on your desktop so that you can edit it. Have the Chemistry Manager or Quality Manager move the file in Qualtrax to the “Archived” folder. Rename the file on your desktop following the same naming convention of the archived document.

3. The “All Drugs Page” of the Excel inventory should have several entries that say “Consumed” or “Disposed” in the first column, along with a date and an analyst’s p#/initials. Open LIMS⇒Resource Manager⇒LVMPD Forensic Laboratory⇒Controlled Substances⇒Reference Materials & Controls⇒Primary/Secondary Reference Materials. In LIMS, locate each consumed or disposed controlled reference material one by one. Double click each lot number to open the Details window. On the right side, there is an Archive check box. Select the check box. In the comments section, type the same information that is in the first column of the Chemical Inventory (e.g. Disposed 11/2/2015 K8702N; Witness M14485N).

4. In the new “Drug Weight History” page on your desktop, delete the columns for these disposed/consumed lot #’s. Save this file, but do not check it back into Qualtrax yet, as it will still need to be edited later. Go back to the “All Drugs” page and delete the same entries.

5. Open the Drug Weight History page that was just archived. In the new “Drug Weight History” page, replace the weight in row 10 with the last recorded weight for each lot# from the archived version, then delete the contents of cell B11 and all the cells to right of and below it. Row 10 now reflects the last recorded weight of the lot# before the archive. Save the changes in the new document and close the archived document.

6. There are several different ways that the inventory list can be sorted/handled (e.g. printing, making an electronic copy, working on the main copy, etc.) to facilitate the audit. Discuss how you want to approach the inventory audit with your audit partner, and find out how other analysts have performed the audit in the past. If you feel comfortable, you can “Hide” the rows that list the non-controlled substances. Be aware, however, that the controlled status for some compounds may have changed between the last inventory and now. If the Inventory list was not kept up to date throughout the year, you will potentially miss some controlled substances.

7. Begin by choosing a physical location in which to start (e.g. CS drawers, refrigerator, freezer desiccator, etc.). It helps if one person handles/weights the reference materials and reads the information to a second person who controls the electronic and/or hardcopy inventory sheets.
8. Go through each reference material one by one and verify that all the fields in the inventory are correct and complete. Change what needs to be changed/updated. This includes, but is not limited to:
   i. Spelling of substance name(s)
   ii. Lot number
   iii. Location Stored
   iv. Container Size
   v. Manufacturer information
   vi. Accuracy and spelling of synonyms
   vii. Quantity on Hand (including tablet/capsule counts)
   viii. Controlled status
   ix. Type of reference material (primary or secondary)
   x. Gross weight of all controlled substances (including tablets or capsules)

9. In addition to verifying the information in all fields of the inventory, ensure that the format of all entries is consistent, and ensure that the storage conditions meet the manufacturer’s guidelines.

10. For controlled substances, all gross weights must be measured and updated in the “Drug Weight History” sheet. All tablets and capsules will be counted, whether they are controlled or not. The tablet/capsule etc. count for all secondary reference materials listed in the “All Drugs” page will be updated for all substances, but only controlled substances need to be updated in the “Drug Weight History” sheet.

11. Verify that all reference materials in the inventory have been accounted for. It is best practice to make a check mark, highlight, or some other notation next to the name of each reference material in the inventory list as you go to ensure that you have accounted for all of them. If there are additional reference materials that are not in the list, check the electronic file to see if you “Hid” that row. If you cannot find the reference material in the list, you will have to add it. If you do not add it to the electronic copy at this time, write down all the pertinent information from the bottle that will need to be added to the inventory at this time (name, manufacturer, lot#, expiration date, product state, container size, quantity, location stored, hazard information, etc.).

12. If any reference material containers have not been marked with an expiration/retest date, add it now (e.g. Exp 11/1/2018). Use a sticker label if necessary. Record the gross weight AFTER adding the sticker and make a note at the bottom of the cell that a sticker label was added in order to account for the weight difference.

13. Record the gross weight with units in row 11 on the “Drug Weight History” sheet. The format for the entry in row 11 is shown in cell A11. List the current date, add your p# and initials, and the gross weight you just measured with units. For the “Used By” line, enter “Inventory.” Use Alt+Enter to move the cursor to the next row within a cell. The contents of this cell should be bolded.

14. Most reference material weights are recorded to (2) decimal places only. Some reference material weights, however, are recorded to (4) decimal places. This is
because the quantity of reference material is so small (1 mg, 5 mg, etc.), that recording the weight to (2) decimal places only records the weight of the container without taking into account the change in quantity inside the container. Most reference materials ordered from Cayman Chemicals have weights that are recorded to (4) decimal places. If you are unsure of how to record the weight, you may ask another analyst or consult the archived Drug Weight History sheet. When in doubt, record to (4) decimal places.

15. If there are any discrepancies in the quantities or weights of controlled substances exceeding ±5% of the previous amount recorded, or if there are missing reference materials, notify the Chemistry Laboratory Manager as soon as possible. This information will be included in a memo summarizing the findings of the inventory.

16. If any reference materials have illegible or incomplete labels, or if any part of the label has been obstructed, create a new label and place it on the bottle(s), or repackage the bottle in a secondary container, labeling it with all the pertinent information. If packaging the reference material in a secondary container, indicate on the container how the gross weight should be measured (with both containers, or only the inner one). Measure all weights after adding the necessary stickers/labels.

17. If any reference materials appear to be discolored, degraded, have compromised packaging, or have any other quality issue, set them aside and discuss the situation with the Chemistry Manager/designee. If the Chemistry Manager/designee approves the disposal, have a second analyst witness the disposal and make a notation in the “All Drugs” page. The notation will be in column A, after the name of the compound, and will be in red font. The notation will read “(Disposed [insert date] [insert P#/initials]; Witnessed by [insert p#/initials]).” Archive these same items in LIMS as well. Also be sure to dispose of any and all GCMS vial stock solutions and/or aliquots in CS Freezer #5 that were made from that lot#.

18. Update the quantity in the “Quantity on Hand” column. In the Drug Weight History sheet, locate the reference material that is to be disposed of. In that column, scroll down the column to the last entry and in the following cell type the p# and initials of the person who disposed of the substance, along with the witness, if applicable, in red font (e.g. R14757A Disposed 3/19/2014; Witness M14485N) and the date.

19. Make a note of any reference materials that could not be located or need to be ordered to replenish the laboratory’s stock.

20. Once the inventory has been completed in its entirety, update the “Inventory Date.” Save and Close the spreadsheet.

21. The Excel Chemistry Inventory should now be up to date and complete. Open Forensic Advantage. Open the Resource Manager module and go to LVMPD Forensic Laboratory → Controlled Substances → Reference Materials & Controls → Primary/Secondary Reference Materials.
22. Check all the information in every controlled reference material LIMS entry against the Chemistry Inventory. The LIMS entries are not required to be as detailed and complete as the Excel Inventory. See the “Procedure for Logging in Reference Materials in LIMS” for the minimum criteria. Archive the lot #’s that were marked consumed/disposed in the Chemistry Inventory, if they have not already been marked as such, including the ones you may have disposed of during this audit. In the “Comments” section, indicate the reason for the archive (e.g. Consumed 5/13/2013 K8702N; Witnessed by M14485N, or Disposed 6/24/2013 K8702N; Witnessed by M14485N, etc.). Update any entries that need to be updated, and add any reference material lot#’s that need to be added. If a folder does not exist for a reference material, contact the Resource Manager Admin for the Chemistry Detail and request that it be added. Be sure to give him/her the name of the compound and whether it is a primary or secondary reference material.

23. Upload the new Drug Weight History page into Qualtrax by creating a new controlled document in the Drug Weight History folder. Seek guidance from the Chemistry Manager, Chemistry Detail, or the Quality Manager if necessary.

24. Inform the Chemistry Manager that you have completed the annual Controlled Substances Inventory Audit.

PROCEDURE FOR NON-CONTROLLED REFERENCE MATERIAL AUDIT

It is not necessary to complete each step in the order presented, but all steps need to be completed.

An annual inventory audit will be conducted on non-controlled reference materials which include non-controlled secondary reference materials and DEA Exempt reference materials. The information maintained in the Chemistry Inventory and the LIMS Resource Manager will be checked and updated.

1. Ask the Chemistry Manager/designee to archive the current version of the Excel Chemistry Inventory by saving a copy in the appropriate “Archive” folder that only Managers and above have access to.

2. The “All Drugs Page” should have several entries that say “Consumed” or “Disposed” in the first column, along with a date and an analyst’s p#/initials. Open LIMS→Resource Manager→LVMPD Forensic Laboratory→Controlled Substances→Reference Materials & Controls→Primary/Secondary Reference Materials. In LIMS, locate each consumed or disposed non-controlled reference material one by one. Double click each lot number to open the Details window. On the right side, there is an Archive check box. Select the check box. In the comments section, type the same information that is in the first column of the Chemical Inventory (e.g. Disposed 11/2/2015 K8702N).

3. There are several different ways that the inventory list can be sorted/handled (e.g. printing, making an electronic copy, working in the main electronic copy, etc.) to facilitate the audit. If two people have been assigned to the non-
controlled inventory audit, discuss how you want to approach the audit with your audit partner, and find out how other analysts have performed the audit in the past. If you feel comfortable, you can “Hide” the rows that list the controlled substances. Be aware, however, that the controlled status for some compounds may have changed between the last inventory and now. If the Inventory list was not kept up to date throughout the year, you will potentially miss some non-controlled substances.

4. Begin by choosing a physical location to start (e.g. NCS drawers, secondary reference materials drawer, refrigerator, freezer desiccator, etc.). It can help if one person handles the reference materials and reads the information to a second person who controls the electronic and/or hardcopy inventory sheets.

5. Go through each reference material one by one and verify that all the fields in the inventory are correct and complete. Change what needs to be changed. This includes, but is not limited to:
   i. Spelling of substance name(s)
   ii. Lot#
   iii. Location Stored
   iv. Container Size
   v. Manufacturer information
   vi. Accuracy and spelling of synonyms
   vii. Quantity on Hand (including tablet/capsule counts)
   viii. Controlled status
   ix. Type of reference material (primary or secondary)

6. In addition to verifying the information in all fields of the inventory, ensure that the format of all entries are consistent, and ensure that the storage conditions meet the manufacturer’s guidelines.

7. Verify that all reference materials in the inventory have been accounted for. It is best practice to make a check mark, highlight, or some other notation next to the name of each reference material in the inventory list as you go to ensure that you have accounted for all of them. If there are additional reference materials that are not on your hard copy list, check the electronic file to see if you “Hid” that row. If you cannot find the reference material in the list, you will have to add it. If you do not add it to the electronic copy at this time, then it is best practice to write down all the pertinent information from the bottle that will need to be added to the inventory at this time (name, manufacturer, lot#, expiration date, product state, container size, quantity, location stored, hazard information, etc.).

8. If any non-controlled primary or non-controlled secondary reference material containers have not been marked with an expiration/retest date, add it now (e.g. Exp 11/24/2015).

9. If any reference materials have illegible or incomplete labels, or if any part of the label has been obstructed, create a new label and place it on the bottle(s), or repackage the bottle in a secondary container, labeling it with all the pertinent information.
10. If any reference materials appear to be discolored, degraded, have compromised packaging, or have any other quality issue, set them aside and discuss the situation with the Chemistry Manager/designee. If the Chemistry Manager/designee approves the disposal, make a notation in the “All Drugs” page. The notation will be in column A, after the name of the compound, and will be in red font. The notation will read “(Disposed [insert date] [insert p# initials]).” Archive these same items in LIMS as well. Also be sure to dispose of any and all GCMS stock solution vials and/or aliquots in CS Freezer #5 that were made from that lot#.

11. Update the “Quantity on Hand” column.

12. Make a note of any non-controlled or secondary reference materials that could not be located or need to be ordered to replenish the laboratory’s stock.

13. Once the inventory has been completed in its entirety, update the “Inventory Date.” Save and Close the spreadsheet.

14. The Excel Chemistry Inventory should now be up to date and complete. Open Forensic Advantage → Resource Manager module → LVMPD Forensic Laboratory → Controlled Substances → Reference Materials & Controls → Primary/Secondary Reference Materials.

15. Check all the information in every non-controlled reference material LIMS entry against the Chemistry Inventory. The LIMS entries are not required to be as detailed and complete as the Excel Inventory. See the “Procedure for Logging in Reference Materials in LIMS” for the minimum criteria. Archive the lot #’s that were marked consumed/disposed in the Chemistry Inventory, if they have not already been marked as such, including the ones you may have disposed of during this audit. In the “Comments” section, indicate the reason for the archive (e.g. Consumed 5/13/2013 K8702N, or Disposed 6/24/2013 K8702N, etc.). Update any entries that need to be updated, and add any reference material lot#’s that need to be added. If a folder does not exist for a reference material, contact the Resource Manager Admin for the Chemistry Detail and request that it be added. Be sure to give him/her the name of the compound and whether it is a primary or secondary reference material.

16. Discuss any discrepancies you found with the Chemistry Manager/designee, including any missing reference materials. A memo will be generated summarizing your findings.

17. Inform the Chemistry Manager/designee that you have completed the Annual Non-Controlled Substances Inventory Audit.
2.3 Title: COLOR TEST REAGENT QUALITY CONTROL

Color test reagents will be assigned an internal lot number and will be quality control checked at the time of preparation (before use on casework samples) using the reference materials designated in the respective recipe. Components of the reagent, along with any reference material’s manufacturer and lot number will be documented in the Resource Manager, or in specific case files for reagents that are for one time use only.

Qualtrax contains the most updated versions of laboratory forms. Most reagents should have their own specialized template on which to document the preparation and quality control checks. For a reagent/solution that does not require quality control checks at regular intervals (with the exception of Marquis reagent), use the General Reagents and Solution Preparation Log. The entries will be completed in accordance with the LVMPD Forensic Laboratory Quality Manual - Chemical/Reagent Issues and Expired Chemicals/Reagents.

In instances where a reagent is prepared for same day use or does not require preparation because it contains only one ingredient, the information required pursuant to the LVMPD Forensic Laboratory Quality Manual must be documented in the case file. Verification that the reagent passes the QC check at the time of case work analysis must be documented in the case record as it pertains to that specific case and therefore does not need to be documented in the Resource Manager.

If at any time the reagent with an assigned internal lot number fails to give the expected result during a QC check, discard the reagent, remake it, and document it in the Reagent Preparation Log in Resource Manager.

The amount of a reagent or solution listed in a recipe found in the Seized Drugs Technical Manual may be scaled up or down to fit the needs of the Chemistry Detail.

When preparing reagents, minor variabilities in measuring liquid volumes do not have a significant effect on the test result. The Controlled Substances Color Test validation demonstrates reproducibility, repeatability, and robustness of reagent preparation, taking into account these minor variabilities in volumes when using existing glassware.

Container Labeling
At a minimum, all laboratory preparations will be labeled with the following information:

- identity of reagent
- lot number
- proper safety warnings
- expiration date
Records shall be maintained in LIMS identifying who made the reagent and the components used in its preparation.

The Marquis reagent will be given an expiration date of **one month**. This reagent will be quality control checked at the time of preparation (prior to being put into use) and again prior to disposal.

The following reagents will be given an expiration date of **one year**. These reagents will be quality control checked at the time of preparation (prior to being put into use), monthly thereafter, and again prior to disposal:

- Chen’s
- 2% Cobalt Thiocyanate
- Modified Duquenois Levine
- Froehde
- Mayer
- Mecke
- Dille-Koppanyi
- Liebermann
- Van Urk
- 5% Ferric Chloride
- Sodium Nitroprusside

If the expiration date of a reagent falls on any scheduled RDO, weekend and/or holiday, the expiration date will be set for the day immediately preceding the RDO, weekend and/or holiday. Reagents should be remade prior to the expiration date.

The following reagents will be made and quality control checked at the time of use. The components used during preparation and the quality control checks will be recorded in the case file. These reagents do not require the assignment of an internal lot number:

- Sulfuric Acid Reagent
- Weber
- Toluene/Cobalt Thiocyanate
- Ammonium Molybdate
- 1% Cobalt Nitrate
- Mandelin
2.4 Title: QUALITY CONTROL PLAN

SAFE HANDLING, USE, TRANSPORTATION AND STORAGE OF EQUIPMENT:
The manufacturer’s Operating or Instruction Manual should be referenced when there are concerns about the handling, usage, transportation and storage of the following equipment.

- Balances
- Non-disposable Pipettes
- GC-FID
- GC-MS
- FTIR-ATR
- XRF
- Benchtop Raman Microscope
- Thermometers
- Portable Raman Devices
- Primary Reference Materials

GC-FIDs, GC-MSs, FTIR-ATRs, XRF, and the Raman Microscope are not transported outside the Forensic Laboratory. Specific balances are used for clandestine laboratory response and remain on the response vehicle when not in use at a scene. The safe handling, use, transportation and storage of these balances will be addressed in the Clandestine Laboratory Response section.

Portable Raman devices will not be typically transported outside the Forensic Laboratory. If the device is transported outside the Forensic Laboratory, it will be placed in its appropriate hard plastic case. The safe handling, use, transportation and storage of the portable Raman devices will follow manufacturer’s specifications.

Non-disposable pipettes will only be transported outside the Forensic Laboratory to an approved external vendor for calibration. During transport, these pipettes will be packaged to minimize potential damage during shipment.

ASTM-1, NIST-F and CS #3 weights will be handled with cotton gloves. Smaller ASTM-1 mass weights will be handled with the plastic tweezers provided by the manufacturer. The weights will be stored in the Chemistry Laboratory in the original manufacturer’s containers and placed in a designated cabinet. The ASTM-1 weights will only be transported outside the Forensic Laboratory to an approved external vendor for calibration. During transport, the weights will be stored in the original manufacturer’s container and packaged to minimize potential damage during shipment.
The NIST-F and CS #3 weight sets will only be transported outside the Forensic Laboratory to perform verifications on the LVMPD balances used by law enforcement officers outside of the Forensic Laboratory. During transport, the weights will be stored in the original manufacturer's container.

Thermometers will be replaced when their calibration certification expires.

Refer to the following table for planned maintenance of selected equipment:

<table>
<thead>
<tr>
<th>Refrigerators/Freezers</th>
<th>Instrument</th>
<th>Frequency</th>
<th>Criteria</th>
<th>Corrective Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS # 5 Frigidaire</td>
<td>Commercial Freezer Model # FFU1724DW2 Ser # WB53137177</td>
<td><strong>External:</strong> None</td>
<td>Fridge: 2-8°C</td>
<td>If a refrigerator/freezer does not meet criteria:</td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>Internal:</strong> Once every two weeks</td>
<td>Freezer: ≤ -15°C</td>
<td>Step 1. Check temperature again within 2 hours</td>
</tr>
<tr>
<td></td>
<td>CS # 6 Sanyo Refrigerator Model # SRR-49GD-MED Ser # KJ00000377M</td>
<td></td>
<td></td>
<td>Step 2. Check thermometer against a second NIST thermometer. Replace if needed, then go directly to step 4. If thermometer is accurate, proceed through remaining steps</td>
</tr>
<tr>
<td>CS # 7 VWR Freezer</td>
<td>Model # HF-5017 Ser # 091200391</td>
<td></td>
<td></td>
<td>Step 3. Adjust thermostat on the unit*</td>
</tr>
<tr>
<td>CS # 8 Frigidaire</td>
<td>Refrigerator/Freezer Model # BA51820809 Ser # FRT17G4BW4</td>
<td></td>
<td>Use “Temperature Check Log” Form found in the Quality folder in Qualtrax</td>
<td>Step 4. If thermometer is inaccurate, replace it directly.</td>
</tr>
</tbody>
</table>

*Note the adjustment(s)
<table>
<thead>
<tr>
<th>Instrument</th>
<th>Frequency</th>
<th>Criteria</th>
<th>Corrective Action</th>
</tr>
</thead>
</table>
| CS #9 Sanyo Refrigerator/Freezer Model # SR-361W Ser # 990869156 | | | that was done**  
Step 4. Monitor and document the temperature within 24 and 48 hours to ensure continued temperature stability  
Step 5. If above steps do not correct the problem, take out of service and advise Seized Drugs Manager / Quality Manager (document on Corrective Action Report if needed)  
*Make gradual adjustment(s). It may take more than one adjustment to find the optimal setting.  
**If temperature deviates more than 3 degrees outside the acceptable range after completing step 3, move refrigerator / freezer contents to an operable unit. If refrigerator / freezer appears to be malfunctioning, immediately move the contents to an operable unit. |
<table>
<thead>
<tr>
<th>Instrument</th>
<th>Frequency</th>
<th>Criteria</th>
<th>Corrective Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pipettes</td>
<td></td>
<td></td>
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<tr>
<td>Pipette identifications and certifications are listed in the Resource Manager</td>
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<td></td>
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<tr>
<td><strong>External:</strong></td>
<td>Annually</td>
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<td></td>
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<tr>
<td><strong>Vendor options:</strong></td>
<td>Calibrate Inc. 1-800-253-7064</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Rice Lake Weighing Systems (800) 472-6703 (925) 798-8900</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Internal:</strong></td>
<td>None</td>
<td></td>
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<tr>
<td>Pipettes not used routinely (stored, clearly marked and used as back-ups) shall have a performance check conducted prior to critical quantitative measurements and then quarterly thereafter if kept in use.</td>
<td></td>
<td></td>
<td>If a pipette is damaged: 1. Tag out of service. 2. Advise Chemistry Manager and Quality Manager/designee who will arrange for repair. 3. Prepare Corrective Action Report, if necessary.</td>
</tr>
<tr>
<td><strong>External:</strong></td>
<td>Meet external vendor specified requirements for calibration criteria.</td>
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<tr>
<td>Vendor certifications are stored in the Resource Manager.</td>
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<tr>
<td>VWR Model # 61161-364 Ser # 180056718 Location: CS #8 Refrigerator</td>
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<tr>
<td><strong>External:</strong></td>
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<tr>
<td><strong>Internal:</strong></td>
<td>None</td>
<td></td>
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<tr>
<td>Replace when calibration certificate expires.</td>
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<tr>
<td>Thermometers must be within ± 1° C of a second NIST certified thermometer.</td>
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<tr>
<td>Certificates of calibration for all thermometers will be stored in the Resource Manager.</td>
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<tr>
<td>Thermometers</td>
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<tr>
<td>VWR Model # 61161-364 Ser # 180056709 Location: CS #5 Freezer</td>
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<tr>
<td><strong>External:</strong></td>
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<td><strong>Internal:</strong></td>
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<tr>
<td>Replace when calibration certificate expires.</td>
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<tr>
<td><strong>External:</strong></td>
<td>Meet external vendor specified requirements for calibration criteria.</td>
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<td></td>
<td>Rice Lake Weighing Systems (800) 472-6703 (925) 798-8900</td>
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<tr>
<td><strong>Internal:</strong></td>
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<td>Replace when calibration certificate expires.</td>
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<td>Criteria</td>
<td>Corrective Action</td>
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<tr>
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<td>Location: CS #9 Refrigerator</td>
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<td>Location: TE Oven</td>
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<tr>
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<td>Ser # 181154685</td>
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<td>Location: Heat Block (Non-critical thermometer)</td>
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<tr>
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<tr>
<td>CS # 16</td>
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<tr>
<td>GC Agilent 7890B</td>
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<tr>
<td># US18013007</td>
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<tr>
<td>MSD Agilent 5977B</td>
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</tr>
<tr>
<td># US1751M012</td>
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</tbody>
</table>

**External:** Refer to specific instrument contract/agreement for appropriate support phone numbers and service schedules.

**Internal:**

**MS Performance (at least weekly):** Perform internal instrument performance check (tune) prior to casework use. At least one internal performance check is needed per week when instrument is used. This performance check must be done the first business day of the week that the instrument is being utilized. The performance check parameters should fall within the “Autotune Guidelines” found in the instrument logbook.

**GC Oven/Column Performance (at least weekly):** Retention times of known reference material checks must be within ± 0.1 minute of the previous run of the checks. Retention times may shift.

At a minimum, attempt the following corrective action if any of the performance checks fail:

1. Repeat test.
2. Troubleshoot using manufacturer’s recommendations.
3. Call for technical support.
4. Tag out of use.
5. Advise lab manager and call for a service engineer.
6. Record problem in the instrument maintenance manual, or, if necessary, on a Corrective Action Report.
<table>
<thead>
<tr>
<th>Instrument</th>
<th>Frequency</th>
<th>Criteria</th>
<th>Corrective Action</th>
</tr>
</thead>
<tbody>
<tr>
<td>CS # 17</td>
<td></td>
<td>more than ± 0.1 minute if instrument maintenance has been recently performed. <strong>Solvent Blanks</strong> (before use) Solvent blanks are required for all solvents used in casework sample preparation. <strong>Maintenance</strong> (as needed) Use “Instrument Maintenance” form found in Qualtrax. Logbooks are located in the lab near the instrument.</td>
<td></td>
</tr>
<tr>
<td>CS # 6</td>
<td>External:</td>
<td></td>
<td>At a minimum, attempt the following corrective action if any of the performance checks fail: 1. Repeat test. 2. Troubleshoot using manufacturer’s recommendations as outlined in the Nicolet 380 User’s Guide. 3. Call for technical support. 4. Tag out of use. 5. Advise lab manager and call for a service engineer. 6. Record problem in the instrument maintenance manual, or, if necessary, on a Corrective Action Report.</td>
</tr>
<tr>
<td></td>
<td>Internal:</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>External: Refer to specific instrument contract/agreement for appropriate support phone numbers and service schedules. <strong>Internal:</strong> Before use</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>Internal: Perform internal instrument performance check prior to casework use. At least one internal performance check is needed per day when instrument is used. Record the minimum/maximum values and pass/fail result of the performance check. Run a known polystyrene sample prior to running casework samples each day the instrument is used. Use “FTIR Performance Check Log” form found in Qualtrax. Logbooks are located in the lab near the instrument.</td>
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<tr>
<td>Instrument</td>
<td>Frequency</td>
<td>Criteria</td>
<td>Corrective Action</td>
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<tr>
<td><strong>CS #12</strong>&lt;br&gt;Raman Microscope&lt;br&gt;Bruker Optics Inc.&lt;br&gt;Model : Senterra&lt;br&gt;Ser # R200.0271</td>
<td><strong>External:</strong>&lt;br&gt;Refer to specific instrument contract/agreement for appropriate support phone numbers and service schedules.&lt;br&gt;&lt;br&gt;<strong>Internal:</strong>&lt;br&gt;Before use</td>
<td><strong>Internal:</strong>&lt;br&gt;Perform internal instrument daily validation prior to casework use. At least one daily validation is needed per day when instrument is used. Print the daily validation test results, sign each page, and place into the logbook. Complete the “Raman Microscope Quality Assurance Log” found in the logbook.&lt;br&gt;&lt;br&gt;Use “Raman Microscope Quality Assurance Log” form found in Qualtrax.&lt;br&gt;&lt;br&gt;Logbook is located in the lab near the instrument.&lt;br&gt;&lt;br&gt;The information is stored electronically and is backed up by Department IT Bureau.</td>
<td>At a minimum, attempt the following corrective action if any of the performance checks fail:  &lt;ol&gt;&lt;li&gt;Repeat test.&lt;/li&gt;&lt;li&gt;Troubleshoot using manufacturer’s recommendations as outlined in the OPUS Users Guide.&lt;/li&gt;&lt;li&gt;Call for technical support&lt;/li&gt;&lt;li&gt;Tag out of use.&lt;/li&gt;&lt;li&gt;Advise lab manager and call for a service engineer.&lt;/li&gt;&lt;li&gt;Record problem in the instrument maintenance manual, or, if necessary, on a Corrective Action Report.&lt;/li&gt;&lt;/ol&gt;</td>
</tr>
<tr>
<td><strong>CS #14</strong>&lt;br&gt;Portable Raman Device&lt;br&gt;Model : TruNarc&lt;br&gt;Ser # TN3379</td>
<td><strong>External:</strong>&lt;br&gt;Refer to specific instrument contract/agreement for appropriate support phone numbers and service schedules.</td>
<td><strong>Internal:</strong>&lt;br&gt;TruNarc:&lt;br&gt;Perform self check with provided self check standard prior to casework use. At least one self check is needed prior to each lab item number. Document the self check scan number and result in case notes.&lt;br&gt;&lt;br&gt;TacticID:&lt;br&gt;Perform performance validation with provided standard prior to casework use. At least one self check is needed prior to each lab item number. The TacticID does not display a scan number (scan index) for the performance validation.</td>
<td>At a minimum, attempt the following corrective action if any of the performance checks fail:  &lt;ol&gt;&lt;li&gt;Repeat self check test and determine if nose cone needs cleaned.&lt;/li&gt;&lt;li&gt;Call for technical support&lt;/li&gt;&lt;li&gt;Tag out of use.&lt;/li&gt;&lt;li&gt;Advise lab manager and call for a service engineer.&lt;/li&gt;&lt;li&gt;Record problem in the instrument maintenance manual located in LIMS, or, if necessary, on a Corrective Action Report.&lt;/li&gt;&lt;/ol&gt;</td>
</tr>
<tr>
<td>Instrument</td>
<td>Frequency</td>
<td>Criteria</td>
<td>Corrective Action</td>
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<tr>
<td>CS #2 XRF, Thermo Scientific Model # Niton XL-3t GOLDD+ Ser # 95039</td>
<td><strong>External:</strong> Refer to specific instrument contract/agreement for appropriate support phone numbers and service schedules. <strong>Internal:</strong> Before use</td>
<td><strong>Internal:</strong> Perform system check and performance check daily before use. The information is stored electronically and is backed up by Department IT Bureau.</td>
<td>At a minimum, attempt the following corrective action if any of the system/performance checks fail: 1. Repeat system check/performance check 2. Call for technical support 3. Tag out of service 4. Advise lab manager and call for a service engineer. 5. Record problem in the instrument maintenance manual, or, if necessary, on a Corrective Action Report.</td>
</tr>
<tr>
<td>CS # 4, Mettler PM 4000 Ser # 216329</td>
<td><strong>External:</strong> Annually <strong>Critical Service</strong> Vendor options: Mettler-Toledo, Inc. 1-800-METTLER Precise Weighing Systems 1-661-250-9044</td>
<td><strong>External:</strong> Refer to Balance Calibration Information chart for specified requirements for calibration located at H:\CB\Forensics\Chemistry\Balances Calibration Information_Seized Drugs <strong>Internal:</strong> Refer to each balance's verification log form located in Qualtrax for acceptable criteria. The determined uncertainty of measurement for each balance is provided to each analyst and can be found in H:\CB\Forensics\Chemistry\UOM. Use appropriate &quot;Balance Verification&quot; form specific to each balance found in Qualtrax. Internal checks are stored in the Resource Manager. Vendor calibration certificates are stored in LIMS under Resource.</td>
<td>If a balance is not operating properly: 1. Initiate manufacturer’s procedures to perform a mechanical internal calibration (if applicable) or external calibration. 2. If the above steps do not correct the problem, tag out of use, advise the lab manager and prepare a Corrective Action Report for balances used for casework.</td>
</tr>
<tr>
<td>Instrument</td>
<td>Frequency</td>
<td>Criteria</td>
<td>Corrective Action</td>
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<tr>
<td>CS #24, Mettler Toledo XS403S Ser # B328537391</td>
<td></td>
<td></td>
<td>Manager.</td>
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<tr>
<td>CS #25, Mettler Toledo XS403S Ser # B328537392</td>
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<tr>
<td>CS #26, Mettler Toledo XS403S Ser # B326493418</td>
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<tr>
<td>CS #27, Mettler Toledo XS403S Ser # B326493429</td>
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<td>CS #28, Mettler Toledo XS80001L Ser # B411395856</td>
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<tr>
<td>CS #29, Mettler Toledo XS303S Ser # B605070460</td>
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<tr>
<td>CS #30, Mettler Toledo XS303S Ser # B605070461</td>
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<tr>
<td>CS #31, Mettler Toledo XS303S Ser # B605070462</td>
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<tr>
<td>CS #22, Ohaus Scout Pro Ser # 7131410764 (Clan Lab)</td>
<td>External: Annually</td>
<td>External: Refer to Balance Calibration Information chart located at H:\CB\Forensics\Chemistry\Balances Calibration Information_Seized Drugs</td>
<td>If a balance is not operating properly: 1. Initiate manufacturer’s procedures to perform a mechanical internal calibration (if applicable) or external calibration. 2. If the above steps do not correct the problem, tag out of use, advise the lab manager and prepare a Corrective Action Report.</td>
</tr>
<tr>
<td>Clan Lab Balances</td>
<td>Critical Service Vendor options: Mettler-Toledo, Inc. 1-800-METTLER Precise Weighing Systems 1-661-250-9044</td>
<td>Internal: Refer to each balance's verification log form located in Qualtrax for acceptable criteria. Internal: Use appropriate “Balance Verification” form specific to each balance found in Qualtrax Internal checks are maintained in the Resource Manager.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Internal: Monthly, field balances used in clandestine lab response – performed with NIST</td>
<td></td>
<td></td>
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<tr>
<td>Instrument</td>
<td>Frequency</td>
<td>Criteria</td>
<td>Corrective Action</td>
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<tr>
<td>certified weight sets.</td>
<td>Per Use: Clan lab balances are to be verified with a mass standard at the time of use.</td>
<td>No uncertainty of measurement was determined for Clan Lab balances</td>
<td>If a weight does not meet the criteria: 1. Take weight out of service 2. Contact the Chemistry Detail Manager/designee and the Quality Manager 3. Prepare a Corrective Action Report, if necessary</td>
</tr>
<tr>
<td>Weight set CS #1 Denver Instruments Ser # N/A (ASTM 1)</td>
<td></td>
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<tr>
<td>Weight set CS #4 Rice Lake Weighing Systems Ser # 5AAX (ASTM 1)</td>
<td><strong>External:</strong> Annually <strong>Critical Service</strong> Rice Lake Weighing Systems (800) 472-6703 (925) 798-8900</td>
<td>Meet external vendor criteria.</td>
<td></td>
</tr>
<tr>
<td>10 pound weight CS #5 Heusser Neweigh Ser # HN6034 Accuracy: NIST F (Used to check MVNC 100 pound scale)</td>
<td><strong>Internal:</strong> None</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Weight Set CS #3 (Used to check balances outside of the Forensic Laboratory e.g. Area Commands, CCDC, etc.)</td>
<td><strong>External:</strong> None</td>
<td>Observed weight must fall within specified criteria on log form. Use &quot;CS Weight Set #3 Verification Log&quot; form found in Qualtrax. Internal verifications are maintained in the Resource Manager.</td>
<td>If the weights do not meet criteria: 1. Repeat test. If the problem is not corrected, tag out of use. 2. Contact the Chemistry Detail Manager/designee and the Quality Manager 3. Prepare a Corrective Action Report, if necessary</td>
</tr>
<tr>
<td><strong>Reference Weights</strong></td>
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<tr>
<td><strong>Field Weight Set</strong></td>
<td></td>
<td></td>
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<tr>
<td>Instrument</td>
<td>Frequency</td>
<td>Criteria</td>
<td>Corrective Action</td>
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<tr>
<td><strong>Clan Lab Field Weights</strong></td>
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<tr>
<td>Clandestine lab weights used to verify field balances used by Clan Lab Response Team</td>
<td></td>
<td><strong>External:</strong> None</td>
<td>If the weights do not meet criteria:</td>
</tr>
<tr>
<td></td>
<td></td>
<td><strong>Internal:</strong> Monthly, performed with NIST certified weight sets.</td>
<td>1. Repeat test.</td>
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<td></td>
<td>If the problem is not corrected, tag out of use.</td>
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<td></td>
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<td></td>
<td>2. Contact the Chemistry Detail Manager/designee and the Quality Manager.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>3. Prepare a Corrective Action Report, if necessary</td>
</tr>
<tr>
<td><strong>Microscopes</strong></td>
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</tr>
<tr>
<td>CS # 2 Fisher Scientific Model: Stereomaster</td>
<td>External:</td>
<td>Meet external vendor criteria</td>
<td>If a microscope is not operating properly:</td>
</tr>
<tr>
<td>Ser # B2608-2128-008</td>
<td>Annually</td>
<td>Vendor service orders are located in Qualtrax</td>
<td>1. Tag out of use</td>
</tr>
<tr>
<td>CS # 3 Nikon Model: PO1 Ser # 66691/15479 Metro # 9176</td>
<td></td>
<td></td>
<td>2. Advise Chemistry Lab Manager to arrange for repair or replacement</td>
</tr>
<tr>
<td>CS # 5 Fisher Scientific Model: Stereomaster</td>
<td>Internal:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ser # A2506-2123-006</td>
<td>None</td>
<td></td>
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<tr>
<td>CS #6 Fisher Scientific Model: Stereomaster</td>
<td></td>
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<tr>
<td>Ser # FW99-209-0004</td>
<td></td>
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<tr>
<td>CS #7 Leica Model: Leica EZ4 Ser # 10447198 958550</td>
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<tr>
<td><strong>Fume Hoods</strong></td>
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<tr>
<td>CS # 3 Model: 7280400FH Ser # 050639185H</td>
<td>External:</td>
<td>Meet external vendor criteria</td>
<td>If a fume hood is not operating properly:</td>
</tr>
<tr>
<td></td>
<td>Annually</td>
<td>Vendor test reports are stored in Resource Manager.</td>
<td>1. Tag out of use</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Internal checks are performed to determine if the fume hoods are operational.</td>
<td>2. Advise Chemistry Lab Manager/designee.</td>
</tr>
<tr>
<td>CS # 4 Model: 7280400FH Ser # 050436147H</td>
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<tr>
<td>CS # 5 Model: 7280400FH Ser # 050639184H</td>
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<tr>
<td>Instrument</td>
<td>Frequency</td>
<td>Criteria</td>
<td>Corrective Action</td>
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</tr>
<tr>
<td>CS # 6</td>
<td>Internal:</td>
<td>Checked monthly during safety checks</td>
<td>If the generator does not meet criteria:</td>
</tr>
<tr>
<td>Model: 7280400FH Ser # 050639182H</td>
<td></td>
<td></td>
<td>1. Tag out of use</td>
</tr>
<tr>
<td></td>
<td>External:</td>
<td>None</td>
<td>2. Troubleshoot using appropriate manufacturer’s manual</td>
</tr>
<tr>
<td>Hydrogen Generators</td>
<td>Internal:</td>
<td>Drained, and deionizer bags are replaced approximately every six (6) months.</td>
<td>3. Advise Chemistry Laboratory Manager</td>
</tr>
<tr>
<td>CS # 2</td>
<td>Internal:</td>
<td>Maintains pressure.</td>
<td>4. Contact manufacturer’s technical support</td>
</tr>
<tr>
<td>Parker-Balston Model: H2-500NA Ser # H2-500514D</td>
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</tr>
<tr>
<td>CS # 3</td>
<td>Internal:</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Parker-Balston Model: H2-500NA Ser # H2-500515D</td>
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<tr>
<td>Instrument</td>
<td>Frequency</td>
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<tr>
<td><strong>Water System</strong></td>
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<tr>
<td>CS #1 ELGA PureLab Flex 3 Model: PF3XXXM1 Ser # FLC00002820</td>
<td><strong>External:</strong> None</td>
<td>Meet manufacturer’s recommendations</td>
<td>If a water purifier system does not meet the criteria:</td>
</tr>
<tr>
<td>SD #1 ELGA PureLab Flex 3 Model #: PF3XXXM1-US Serial #: FLC00010871</td>
<td><strong>Internal:</strong> Purification Filter – replace every 6 -12 months or as needed Pretreatment Filter – replace approximately every 6 months or as needed Vent Filter – replace approximately every 12 months or as needed UV lamp – replace approximately every 18 months or as needed Quartz thimble – replace approximately every 18 months or as needed, typically done with UV lamp</td>
<td></td>
<td>1. Take out of service 2. Replace filter(s) and/or UV lamp as necessary 3. Advise Chemistry Laboratory Manager/designee 4. Contact ELGA technical support</td>
</tr>
<tr>
<td></td>
<td>For repairs and maintenance: ELGA (877) 315-3542</td>
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</table>
LVMPD FORENSIC LABORATORY
TECHNICAL PROCEDURES
SEIZED DRUGS

3.1 Title: SEIZED DRUGS ANALYSIS QUALITY CONTROL

EVIDENCE INSPECTION AND HANDLING
Upon receipt of seized drug evidence, the assigned Forensic Scientist shall store the evidence in the Chemistry Detail secured evidence vault until the time of analysis. Prior to analysis, the Forensic Scientist shall visually inspect all package(s) to ensure that:

1) The evidence tape is intact and properly sealed
2) The packaging doesn’t appear compromised in any way
3) Each evidence package displays the appropriate event/case # and item #s

Minor tears/holes in the packaging can be reinforced with clear tape and labeled with the analyst’s p#/initials and date. Evidence tape that has started to peel away from the packaging can also be remedied in this manner, so long as items could not have been removed/added to the package as a result of the loss of seal.

Major anomalies noted with any of the above characteristics should be reported to the Forensic Laboratory Evidence Technicians for resolution.

The contents of every package opened will be inventoried by comparing the contents to the evidence label and laboratory request. Refer to the Evidence Discrepancies chapter if a discrepancy is noted.

The visual examination and physical characterization of the evidence shall be recorded in the worksheet, and should include where applicable:

- Type of material (e.g. powder, liquid, tablet, plant, etc.)
- Color
- Size, Shape
- Morphology
- Significant markings (e.g. pharmaceutical markings, score marks, etc.)

During analysis, all examined evidence will be labeled with a unique identifier. At a minimum, all proximal containers (the container closest to the evidence) or the item of evidence itself will be labeled with the agency event/case number, lab number, lab item number, and Forensic Scientist’s P# and initials.

The creation of sub-sub-items will be at the discretion of the analyst and will follow the sequential dot number format. Examples: 1.3.1; 1.3.2; 1.3.3 OR 1.4.1.1; 1.4.1.2; 1.4.1.3, etc.

Precautions shall be taken to avoid deterioration, contamination, loss or damage to the evidence during handling, transporting, storing, and preparation for testing. Specified environmental conditions (e.g. refrigerate, freeze, etc.) for evidence shall be maintained while in the analyst’s custody.
If necessary, place sharp objects (needles, syringes, broken glass) into a puncture-resistant container. Make note of repackaging in case notes. Attach any warning labels to the outside packaging if applicable (e.g. biohazard, broken glass, etc.).

The following items are not routinely analyzed, however, special circumstances may require analysis and must be authorized by the Chemistry Laboratory Manager/designee or Director of Laboratory Services.

- Syringes, when other suspected drug items are in the case
- Currency
- Drug paraphernalia
- Residues

Evidence that is received as compressed material (commonly referred to as a brick or a bundle) should be broken to ensure reasonable homogeneity.

Refer to Chapter 3.7 for samples that are suspected to contain fentanyl or fentanyl related substances.

**DOCUMENTATION:**
Original observations, data and calculations shall be recorded contemporaneously at the time they are made. Observations could include, but are not limited to, written notes, photography, drawing, photocopying, or scanning.

Notes can be taken on form LVMPD ISD 27 or electronic form LVMPD ISD 27a if the LIMS is not operational. The case notes completed outside of LIMS will be uploaded as an object in the Unit Record Object Repository once LIMS functionality has been restored. Refer to the Forensic Laboratory Quality Manual.

Notes must be legible, in permanent medium, and complete, for they often are referred to months and even years after the analysis was performed.

Test information and/or results will never be deleted from case notes. If a test is repeated any number of times, every additional test(s) must be documented in the case notes. Results of presumptive tests for which reviewable data does not exist shall not be altered without an explanatory note added to the case notes.

Proper notes shall include the following:

- Lab case and lab item number
- Description of evidence and packaging
- Any packaging problems or evidence oddities/discrepancies
- Name of test(s) performed and result(s)
- Results and conclusions upon completion of analysis
- Quality control checks, if applicable
- Whether static or dynamic weighing was used, if applicable
- An indication that additional packaging was introduced, if applicable
- Environmental conditions (e.g. refrigerated, frozen) under which the evidence was stored while in the analyst’s custody, when necessary. The unique identifier of the particular refrigerator/freezer along with associated timeframe shall be documented.
Communication regarding the case must be maintained in the appropriate Communication Log or uploaded to the appropriate Object Repository.

**Technical Records**

Notes and all case data (e.g. spectra, chromatograms, photocopies [of literature], prints [of internet pages], photographs, and sketches) must remain in the case record, and each page shall bear the associated lab case number. All data will also bear the unique instrument number from which that data was acquired. For supplemental analyses, each page of the technical record shall also indicate that it was created during the supplemental analysis (e.g. add “S” or “Supp” to the title).

All files uploaded to the Object Repository will display the lab case number in the title of the file.

Electronic copies of the case data (e.g. spectra, chromatograms, photocopies [of literature], prints [of internet pages], photographs, and sketches) will be deleted immediately after the final version is included in the Object Repository. This does not include the raw instrument data.

In addition to the above listed requirements, if a photocopy is made from any hardcopy literature reference source and included in the case file, then, at a minimum, the title of the literature reference source and the version (e.g. 4th edition) shall be noted on every photocopied page. These documents will be uploaded to the Unit Record Object Repository.

**Use of Photography for Case Documentation**

Photographs should be taken by the analyst to document:

- unusual situations
- discrepancies
- significant changes made by an analyst to a submitted item of evidence
- instances when the evidence item(s) are consumed during analysis

At a minimum, each photograph must have the analyst’s P#/initials, lab case and lab item number. These annotations are not considered alterations to the photograph(s).

If photographs were not taken when one of the above listed situations exists, an explanation of why photographs were not taken shall be included in the case notes. It is recognized that photography is primarily for recording purposes, and it is inherently limited in its ability to record all of the observed detail.

Digital photography and electronic image capture and processing can be used in the same way standard photography is used. Images will not be altered in any way with the purpose of misrepresenting the evidence. If the image is altered, the original image must be saved along with the altered image. These digital image files will be retained as electronic files and uploaded to the Object Repository.

**WEIGHTS AND QUANTITIES:**

Balances should always be clean and tared prior to any sample weighing.
Weights and quantities of materials submitted for analysis will be measured prior to sampling and will be recorded on the Seized Drugs worksheet. If the weights or quantities being reported were measured after sampling (e.g. a sample was subsequently split into sub-items, etc.), it will be clearly indicated in the notes. It is preferred that the gross weight of the evidence be reported. When the gross weight is reported, the description of the item on the report shall include all packaging that was weighed. The net weight of the material may be measured at the discretion of the analyst.

If the gross weight of the item(s) is within close proximity of an established weight threshold as defined in the Nevada Revised Statute (NRS) 453 or United States Sentencing Guidelines (for Federal cases), the net weight of a sufficient number of items shall be reported.

The minimum load for each balance is twice the uncertainty of measurement (UOM) determined for that particular balance. For example, if the UOM for balance CS #9 is +0.03 g, the minimum load for this balance would be 0.06 g. Actual balance readings as they are displayed on the balance are recorded, not rounded. If the total weight is below 0.05 g for an item, the weight(s) will be recorded in the notes, but reported as “residue”.

If the estimated UOM for the balance used is equal to or greater than the weight measured, a more accurate balance shall be used, or the substance shall be reported as a residue, whichever is appropriate. Weigh boats should not be used for measuring weights on 4-place and 5-place balances.

When multiple balances are used to record weights of units within one item, the sum of the weights recorded with each balance shall be reported separately.

Pharmaceutical or illicit tablets, tablet fragments, pills, and/or capsules must be assessed for homogeneity and weighed, when being analyzed. Counting is only required if hypergeometric statistical sampling will be used. The piece counting feature on analytical balances will not be used to determine the total count.

Cigarettes and perforated paper squares suspected to contain a controlled substance (e.g. marijuana, PCP, and LSD) will be counted and weighed, when being analyzed. The weight and number of items shall be reported.

For Federal cases involving analysis of suspected LSD, perforated paper squares will be considered single dosage units and will be subjected to an approved sampling plan. Special cases (e.g. non-perforated paper) will be handled on a case by case basis at the discretion of the analyst.

Approximate volumes may be recorded in the notes, but will not be reported (with the exception of certain clandestine laboratory analyses).
REAGENTS:
QC checks of reagents will be performed at the time of preparation and prior to disposal (excluding one-time use reagents) to verify that the reagent is working as expected. Reagents and solvent systems used for TLC are QC checked concurrently with use because the reference material(s) is run on the same plate every time. Refer to the Recipes and Derivatizing Agents chapter for procedures.

Reagents shall be discarded if there are indications of instability (such as discoloration).

Expired reagents and chemicals will not be used for casework.

INSTRUMENTATION:
Instruments are routinely tuned and/or performance checked to ensure operation is acceptable and meets the manufacturer’s specifications.

An instrument log book is maintained to document all repairs, solvent checks, column test check runs, performance checks, and maintenance.

Instrument parameters for each method will be stored in Qualtrax.

GC-FID and GC-MS
Dated tune and column test check data must be approved by an analyst before utilizing the instruments for casework and will be maintained electronically. It is each analyst’s responsibility to ensure that an acceptable tune and column test check have been run for the GC-MS instrument for that week prior to running his/her casework samples. Ensure the tune report and column test check run are stored in the instrument’s logbook or electronically.

When a large GC-MS sample run sequence continues over multiple days, the run sequence does not need to be interrupted to tune the instrument or run the column test checks (if applicable). The tune and column test checks (if applicable) used to ensure the proper operation of the instrument on the day the sequence began will suffice for the duration and completion of the run sequence. The instrument will be tuned and the column test checks run (if applicable) after the said run sequence has completed, before any additional casework samples are analyzed.

All sample vials will be clearly marked with a unique identifier created by the analyst.

The sequence table will be checked against the vials in the autosampler tray prior to starting the autosample run.

Retention times must be compared to a reference material run on the same analytical method and instrument. If retention data is used as a screening test to support the identification of an unknown, the “Retention Time Checked” box will be checked on the worksheet. If retention data is not used, the “Retention Time Not Checked” box will be checked and a reason will be included. Retention time data will be maintained electronically on the instrument computer.
Blanks and the samples they precede must be prepared using the same extraction scheme and with solvents/chemicals of the same manufacturer and lot number. Blanks will be processed through the same equipment in a manner similar to the sample e.g. test tube caps, swabs, filter paper, etc.

When samples from one case are interrupted with samples from a different case, an additional blank must be run prior to resuming analysis of the remaining samples from the original case. The blank data will be included in each case file.

At least one page of each data file must be marked in a way that shows that all peaks in the total ion chromatogram (TIC) have been checked.

At times it may be beneficial to use the GC/MS instrument software to subtract out background ions for a mass spectrum. Background subtraction should be done sparingly and only with the approval of the Chemistry Laboratory Manager/designee. If background subtraction is used, the case file will include the original mass spectrum, the subtracted mass spectrum and the resultant mass spectrum, all clearly labeled.

All casework samples will be compared to a verified reference material or a reference library for a conclusive identification. All spectra used for identification will conform to the acceptance criteria listed in chapter 1.4.

In the presence of an identifiable controlled substance, subsequent identification and reporting of minor constituents (weak samples/common cutting agents) is not required when the initial data generated does not meet the identification criteria for the technique. Substances should be confirmed within reason, however, is not required if the instrument must be overloaded with the major constituent.

$\Delta 9$-THC or $\Delta 9$-THCA will not be reported unless a technique that can differentiate the two has been used (e.g. TLC, derivatization, etc.).

If no controlled substance(s) and/or dangerous drug(s) can be identified, at least one (1) sample from each item shall be run on a more general method (e.g. RUN_C or RUN_CL).

**FTIR and FTIR-ATR**
It is each analyst’s responsibility to ensure that the instrument has been properly qualified prior to running casework samples. Ensure that the qualification report and tests are stored in the instrument logbook or electronically.

A background spectrum will be run prior to running samples for each case. When using the ATR attachment, a background is obtained by keeping the anvil in a raised position and choosing ‘Collect Background’ from the software menu. When using KBr pellets, a background is obtained by choosing ‘Collect Background’ from the software menu when the analysis chamber is empty, but covered.

When using the ATR attachment, the diamond cell and anvil will be cleaned thoroughly after each sample to prevent carry over and contamination.
All backgrounds and sample data will be included in the case file. If KBr is used for sample preparation, then a blank KBr pellet must be run prior to the sample (e.g. background, KBr blank, KBr case sample).

All casework samples will be compared to a verified reference material or a reference library for a conclusive identification. All spectra used for identification will conform to the acceptance criteria listed in chapter 1.3.

**XRF**

It is each analyst’s responsibility to ensure that the instrument has been properly performance checked prior to running his/her casework samples.

Samples will be placed in manufacturer approved, clean, disposable XRF sample cups and covered with a clean disposable plastic film. *Both the sample cups and plastic film are one time use only.*

**UNUSED INSTRUMENT DATA FILES**

At least one (1) page from each unused data file shall be included in the case file. The initials of the analyst, lab case number, date, and a note as to why the data was not used must be noted on the electronic data page(s). All files bearing the lab case number shall be included in the case file, and no files bearing the lab case number will be overwritten or deleted.

**BACKING UP DATA FILES**

The data from all the GC-MS and FTIR instruments is backed up by the LVMPD IT Bureau. XRF and Raman Microscope raw data files will be stored in the case file.
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3.2 Title: SAMPLING

Sampling is taking a part of a substance, material or product for testing in order to make an inference about, reach a conclusion, and/or report on the entire substance. One must be sure that what is sampled is truly representative of the total population. The analyst must take the homogeneity of each item into consideration. Careful visual inspections and personal experience are essential in determining the proper sampling procedure.

The sampling date(s) shall be documented in the case file.

Statistical sampling should only be used when there is a reasonable expectation of homogeneity of the entire substance. For items containing multiple units, a statistically-based hypergeometric sampling plan allows the Forensic Scientist to analyze a portion of the items in order to make a statistical inference about the larger population.

Sample selection is a practice of selecting units of items to test based on training, experience, and competence. There is no expectation of homogeneity when using sample selection. A fixed number of items within a population may be analyzed, however an inference to the entire population will not be drawn. The number of items that were analyzed will be clearly indicated in the case notes and on the report.

Under no circumstances will extractions from multiple units be combined and analyzed as one.

If identical intact, marked pharmaceutical preparations (e.g., tablets or untampered capsules) are present as multiple items, analysis is required for only one item. Those preparations not analyzed may be reported as “Received, not examined”.
STATISTICAL SAMPLING CRITERIA:

- The Seized Drugs Unit uses the hypergeometric statistical sampling plan with an approximate 95% confidence level to determine the number of units to be tested. Refer to Table 1 for the number of units requiring conclusive testing. Application of the hypergeometric statistical sampling plan is based on a reasonable assessment of homogeneity and the random sampling of units.
- Random sampling ensures that all units in a population have an equal chance of being selected, and selection bias is avoided.
- If individually packaged items appear homogeneous, a random sample of the units will be selected for analysis. The total number of like pharmaceutical preparations such as tablets and capsules can be subjected to random sampling regardless of packaging.
- All randomly selected units will be conclusively analyzed.
- Contents of separate packages/containers will not be physically combined with each other.
- If all randomly selected units do not contain the same controlled substance(s) all units must be analyzed, or sample selection may be performed. Controlled substance(s) should be confirmed within reason, however, it is not required if the instrument must be overloaded with the major constituent.
- If all randomly selected units contain the same controlled substance(s), but some of them contain a substance that was not controlled at the time of booking, it is not necessary to analyze all units (report only the controlled substance(s) that was identified in all randomly selected units). If any of the randomly selected units are found to contain an identifiable substance (controlled or non-controlled) while the remainder of the randomly selected units are completely negative for both controlled and non-controlled substances, all units must be analyzed, or sample selection may be performed.

SAMPLE SELECTION CRITERIA:

- A limited number of units will be conclusively tested, however, an inference to the entire population will not be made.
- When sample selection (or a non-statistical sampling plan) is used, the report must state what was received, what was tested, and must be clear that the results pertain only to that which was tested.
- The weight of the entire item must be reported in the “Description” portion of the report, and the weight of the units actually tested must be reported in the “Results, Opinions, and Interpretations” portion of the report.

A limited analysis for Schedule II controlled substances submitted in a commercially manufactured form may be performed. Analysis of one (1) tablet/pill/capsule is sufficient for sample selection testing, provided that the instrumental results agree with the reference source comparison.

Schedule III – V controlled substances in Nevada are charged as possession of a controlled substance (PCS) only and do not have statutory threshold levels. Analysis of only one (1) tablet/pill/capsule is sufficient.
Table 1: Statistical Sampling Plan

<table>
<thead>
<tr>
<th>Total Number of Units</th>
<th>Minimum Number of Units to Analyze</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt;10</td>
<td>All</td>
</tr>
<tr>
<td>10-12</td>
<td>9</td>
</tr>
<tr>
<td>13</td>
<td>10</td>
</tr>
<tr>
<td>14</td>
<td>11</td>
</tr>
<tr>
<td>15-16</td>
<td>12</td>
</tr>
<tr>
<td>17</td>
<td>13</td>
</tr>
<tr>
<td>18</td>
<td>14</td>
</tr>
<tr>
<td>19-24</td>
<td>15</td>
</tr>
<tr>
<td>25-26</td>
<td>16</td>
</tr>
<tr>
<td>27</td>
<td>17</td>
</tr>
<tr>
<td>28-35</td>
<td>18</td>
</tr>
<tr>
<td>36-37</td>
<td>19</td>
</tr>
<tr>
<td>38-46</td>
<td>20</td>
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<tr>
<td>47-48</td>
<td>21</td>
</tr>
<tr>
<td>49-58</td>
<td>22</td>
</tr>
<tr>
<td>59-77</td>
<td>23</td>
</tr>
<tr>
<td>78-88</td>
<td>24</td>
</tr>
<tr>
<td>89-118</td>
<td>25</td>
</tr>
<tr>
<td>119-178</td>
<td>26</td>
</tr>
<tr>
<td>179-298</td>
<td>27</td>
</tr>
<tr>
<td>299-1600</td>
<td>28</td>
</tr>
<tr>
<td>More than 1600</td>
<td>29</td>
</tr>
</tbody>
</table>

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3.3 Title: IDENTIFICATION CRITERIA

REFERENCE SOURCE CRITERIA:
A reference source comparison requires the comparison of physical characteristics of a commercially prepared pharmaceutical to the description/depiction contained in a literature reference source.

Pharmaceutical preparations are visually examined using pharmaceutical identifiers and appropriate references.

References are valuable tools to help identify the contents of legitimate pharmaceutical preparations, and include but are not limited to:

- Drugs.com
- The Physician’s Desk Reference
- Pillbox by National Library of Medicine, National Institute of Health
- Amera-Chem Inc. (RxID)
- Drug Identification Bible
- Anabolics Reference Manual
- The National Drug Code Directory

Pharmaceutical preparations that are coded to contain only non-controlled substances can be analyzed using reference source examination only. The report will clearly state that the analysis was based on a visual comparison of the item’s characteristics to a literature reference.

If the analyst has reason to believe that a non-controlled pharmaceutical preparation may be counterfeit, at least one unit must be subjected to conclusive identification criteria.

If the literature reference source(s) provide contradictory information for any pharmaceutical preparation (e.g. the literature reference search results show two different possibilities for what the preparation contains), at least one unit must be subjected to conclusive identification criteria.

If the instrumental data indicates that the preparation is inconsistent with the literature source information or is found to be counterfeit (e.g. literature reference source indicates that the sample contains hydrocodone but analysis on the GC-MS shows the presence of oxycodone), at least one unit must be subjected to conclusive identification criteria.

Cases may require conclusive testing for the purposes of court.
CONCLUSIVE IDENTIFICATION CRITERIA:
When a validated Category A technique is incorporated into an analytical scheme, at least one other technique (from either Category A, B or C) shall be used.

When a Category A technique is not used, at least three different validated techniques shall be employed. Two of the three techniques shall be based on uncorrelated techniques from Category B.

<table>
<thead>
<tr>
<th>Category A</th>
<th>Category B</th>
<th>Category C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Infrared Spectroscopy</td>
<td>Gas Chromatography</td>
<td>Color Tests</td>
</tr>
<tr>
<td>Mass Spectrometry</td>
<td>Marijuana only:</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Macroscopic Examination</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Microscopic Examination</td>
<td></td>
</tr>
<tr>
<td>Raman Spectroscopy</td>
<td>Microcrystalline Tests</td>
<td></td>
</tr>
<tr>
<td>(benchtop)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>X-ray Fluorescence</td>
<td>Pharmaceutical Identifiers</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Portable Raman Spectroscopy</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Thin Layer Chromatography</td>
<td></td>
</tr>
</tbody>
</table>

For marijuana, macroscopic and microscopic examinations are considered uncorrelated techniques from Category B.
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3.4 Title: EVIDENCE DISCREPANCIES AND PRELIMINARY FIELD TEST ERRORS

Review the LVMPD Forensic Laboratory Quality Manual policy regarding Evidence Discrepancies.

DISCREPANCIES GREATER THAN ±10% AND MISSING ITEMS:
When an unexplained discrepancy greater than ±10% (either in count, weight, etc.) exists between the actual contents/weights present and the description on the evidence label, or items are missing, the analyst will email the Chemistry Laboratory Manager/designee, complete the CS Discrepancy Log, and document the discrepancy in the case notes.

Photographs are optional, however, any photographs that are taken must be uploaded to the appropriate Object Repository. All photographs taken must include:
- The outside of the package including the evidence label and seals
- The affected items of the package
- In the case of missing items, all items received
- The weight measured in the laboratory, if applicable
- Other photographs necessary to clearly document the discrepancy

The email to the Chemistry Manager/designee must include an explanation of the discrepancy and a reference to the lab case #(s) and lab item #(#(s) affected. Also include whether photographs were taken. If photographs were taken, ensure the Manager/designee can access them in the Object Repository prior to sending the email.

The case notes will reflect how the discrepancy was documented (e.g. witnessed by Manager, another analyst, photographs taken, etc.).

The CS Discrepancy Log is located at: H:\CB\Forensics\Chemistry\Controlled Sub\CS discrepancy log

If a missing item is discovered, analysis must be stopped and the evidence package(s) must be returned to the Evidence Vault. The missing item(s) will be documented in the case notes. Refer to the Forensic Laboratory Quality Manual for additional requirements.

DISCREPANCIES LESS THAN ±10%:
Discrepancies less than ±10% (either in count, weight, etc.) do not typically require additional documentation, but may still be photographed and documented at the analyst’s discretion.
ADDITIONAL CIRCUMSTANCES:
There are times when discrepancies may have a plausible explanation. Examples include:

- Water loss from plant material
- Officer weights <1 gram may be difficult to measure depending on the readability of the balance used by the officer
- Net weight might have been marked instead of gross weight, or vice versa
- The balance used may have been set to ounces instead of grams
- A missing item located inside another package in the case
- An extra item in the package may be listed on another package in the case

In situations where moisture was lost from plant material or weights <1 gram were recorded by the officer, additional documentation in the case file is not typically necessary.

If it is believed that the officer marked the wrong type of weight measured (net vs. gross), a reference to the type of weighing the officer likely used should be made in the case notes.

If it is believed that the officer had the balance set to the wrong units, sufficient notes shall be added to the worksheet as supporting documentation (e.g. conversion factors, calculations, etc.).

Extra drug items not listed on the evidence label of the package shall be documented in the case notes, and photographs showing what was received may be taken and uploaded to the Unit Record Object Repository. After analysis is complete of the requested of the item(s), the Evidence Technicians shall be notified of the extra item(s) upon return of evidence.

PRELIMINARY FIELD TEST ERRORS:
Per Department Policy 5/210.32 Preliminary Field Narcotic Test For Controlled Substances, when a field test checklist is incorrectly completed or has resulted in a false (+), an error memo is sent to the officer’s chain of command. Error memos are only sent for LVMPD cases.

A proper and complete checklist should have the following fields completed correctly:

- Event#
- Package and item # of the item tested
- Officer’s name and/or p#
- Date the officer was certified
- Date the preliminary test was performed
- Weight, with either “net” or “gross” checked (see additional information below)
- The lot# of the kit(s) used, unless there is no lot# field on the checklist
- Serial # of device, if applicable
- All appropriate boxes checked and/or scan numbers entered for each test that was performed
- Officer’s signature
- Witness signature (except handheld Raman checklists which do not require a witness)

"Net" or "Gross" do not need to be checked if the weight listed is < 0.1 g or if the item was requested for analysis, as the officer's weight is not typically used for filing charges. These instances should still be documented in the worksheet.

Allowances can be made if the package and item number are not complete, as long as the analyst can reasonably conclude which item the checklist corresponds to (e.g. it’s the only suspected drug item in the case). However, if the request for analysis was received because the requester indicated that the checklist was completed incorrectly, an error memo should be sent, as this indicates that the information provided on the checklist was not sufficient for court purposes.

When the preliminary field test checklist inside the evidence package has been incorrectly completed:
- Document the details of the error in the worksheet. Include:
  - The result of the field test as indicated by the officer on the checklist
  - The error(s)
  - The officer’s P#/initials or P#/name
- Scan the checklist (or make a hard copy) and send to the LEST in charge of creating error memos. Because the scans/copies can be difficult to read, include the following information when submitting the form to the LEST:
  - The lab case # and event #
  - The error(s)
  - The officer’s name and/or P#
  - Analyst P#/initials and/or name

If the checklist corresponds to an item that has been requested for analysis, continue with analysis.
If the checklist corresponds to an item that has not been requested for analysis, the analyst should notify the requester to determine if analysis on that item should be performed. All communications will be documented in the Case Communication Log or uploaded to the appropriate Object Repository.

When laboratory testing indicates that the preliminary field test has resulted in a false (+):
- Scan the checklist (or make a hard copy) and send to the Chemistry Laboratory Manager and the LEST in charge of creating error memos. Because the scans/copies can be difficult to read, include the following information when submitting the form:
  - The lab# and event#
  - That the test resulted in a false (+), along with any other error(s) noted
  - The officer’s name and/or P#
  - The result after laboratory analysis
  - Analyst P#/initials and/or name
- Perform the same preliminary field test in the laboratory that the officer used in the field
o When using a color field test kits, have another analyst witness the performance of the test
o Follow the step by step instructions on the checklist. Stop testing if the result is ‘inconclusive’ at any point
o Document the name of the test, the lot# of the kit/serial# of device, the results, and who the witness was in the worksheet
o If the laboratory does not have the appropriate kit in-house, notify the Chemistry Laboratory Manager/designee and try to obtain the kit from the LVMPD Supply section. If the kit will not be received in a timely manner, document that in the worksheet as the reason for not performing the test in-house
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3.5 Title: MARIJUANA ANALYSIS

Plant material suspected of being marijuana is generally examined using either three (3) category B techniques, or two (2) uncorrelated techniques from category B and one (1) technique from category C. If those criteria cannot be met, a category A technique may be used, however the result will be reported as “Tetrahydrocannabinol” not “Marijuana”.

All observations must be documented contemporaneously at the time of the test.

- Macroscopic characteristics include, but are not limited to, observing flowering tops or “buds,” serrated and/or palmate leaves that appear curled when dried, leaves with one darker green side and one lighter green side, and/or resinous extract visible as clear/brown droplets. Recorded observations must include whether the sample is consistent or inconsistent with marijuana, but may also include further details about what was observed.

- At a minimum, microscopic examination involves observing the sample under a stereomicroscope for the presence of cystolithic hairs and simple/covering hairs on opposite sides of the same leaf. The presence or absence of these hairs on opposite sides of the same leaf indicates that the sample is either consistent or inconsistent with marijuana. Other characteristics observed may also be recorded.

- The Modified Duquenois-Levine (or Rapid Modified Duquenois-Levine) test is a chemical color test that results in a purple/lavender color that extracts from the upper layer into the chloroform lower layer in the presence of cannabinoids. A blue-purple over purple result is considered a positive test, however it is understood that several factors including, but not limited to, the age of the plant, resin content, prior processing, matrix interferences, etc. can all affect the shade and the hue of the color observed.

- Thin Layer Chromatography (TLC) provides a separation technique that allows for the differentiation of THC and THCA. Therefore, any conclusions of “Tetrahydrocannabinol” that are supported by GC-MS must also be supported by TLC (or derivatization). The appropriate reference material(s) must be run on the same plate as the sample in order to compare the relative distance travelled and the stained color.

The case notes shall clearly indicate how many samples were tested per item. The case notes can reflect either the number of samples tested or which specific samples were tested. For example: If microscopic examination was performed on nine (9) of the twelve (12) samples, the result can be written as “Microscopic: (+) MJ – covering and cystolithic hairs present x 9” or as “Microscopic: (+) MJ – covering and cystolithic hairs
present (1)-(9).” If only one unit in the item is being tested, it is not necessary to indicate the number of units tested. Documenting the result is sufficient and it is implied that only one (1) sample was tested.

If the same test is repeated on a sample, then an additional test entry must be added to the case notes. This additional test indicates that the same test was repeated on this particular item a second time. For example, the Modified Duquenois-Levine color test was performed two (2) different times on the same item, which consists of six (6) packages. The case notes will reflect:

```
Modified Duquenois-Levine:
Duquenois Levine [C052616-1] Straw x 6 or Straw (1)-(6)
Hydrochloric Acid – 2.5L Purple x 6
Chloroform – 1L Purple over purple x 6
```

```
Modified Duquenois-Levine:
Duquenois Levine [C052616-1] Straw x 6 or Straw (1)-(6)
Hydrochloric Acid – 2.5L Purple x 6
Chloroform – 1L Purple over purple x 6
```

Evidence containing only suspected marijuana seeds and stems are not typically analyzed. Authorization from the Chemistry Laboratory Manager/designee or the Director of Laboratory Services must be obtained before performing viability tests.

Marijuana plants should not be counted during analysis.

The gross weight for bulk marijuana samples should be reported. The laboratory report must include the packaging (e.g. box, paperbag, etc.) that was included in the weight in the description.

Federal cases involving suspected hashish and/or hashish oil require additional reporting. Any and all of the following compounds that were conclusively identified must be reported:

- THC isomers listed in 21 C.F.R. § 1308.11(d)(31)
- Cannabinol
- Cannabidiol
- Cannabichromene
3.6 Title: OPIUM ANALYSIS

Opium is generally encountered in one of four recognized forms: raw opium, prepared opium, opium dross, and medicinal opium.

Raw opium is dried opium latex. It will always contain some quantity of plant fragments as a natural outcome of the harvesting process, and can be cut with, among others things, flour, soil, rosin, or banana pulp. When fresh, it has a tar-like consistency, is very sticky, and is usually a medium brown in color. As raw opium ages, it will gradually become hard and brittle, and the color will become darker, especially at the surface. Distinguishing features of raw opium are its characteristic odor, the presence of plant fragments, and the presence of meconic acid and the porphyroxines.

Prepared opium (also known as cooked opium) is often produced by dissolving raw opium in hot water, filtering to remove the insoluble materials, and evaporating until the filtrate again becomes a solid paste. This material is prepared almost exclusively for smoking purposes.

Opium dross is the residue left after opium has been smoked. There are many local names for dross. For instance, in much of Southeast Asia it is known as "chandu," while in Iran it is known as "sukhteh." Dross is eaten or re-smoked after being added to prepared opium. The presence of dross in prepared opium is generally obvious as a charred material within the prepared opium, and a "burnt" odor is frequently present.

Medicinal opium is generally one of three preparations. The first is "granulated" or "powdered" opium (depending upon the final mesh size of the product), and is an opium which has been thoroughly dried at 70° C and diluted with lactose to give a morphine content between 10 and 10.5% by weight. A second medicinal opium is known as either "deodorized opium" or "denarcotized opium." This material is prepared by treating opium with petroleum ether, which removes both narcotine (noscapine) and the characteristic odor of opium. The concentration of morphine in denarcotized opium is also 10 to 10.5% by weight. The third medicinal opium has been modified rather extensively, and is commonly known within the U.S. under the trade name of Pantopon. This preparation is also known as "concentrated opium," Omnopon, or perhaps most commonly, Papaveretum. It is a mixture of morphine, codeine, papaverine, and noscapine as hydrochloride salts, with the morphine content adjusted to approximately 50% by weight.

MICROSCOPIC EXAMINATION:
Plant debris can be isolated for microscopic examination by exhaustively washing the opium with water. The residue will contain poppy capsule fragments and occasionally spherical pollen grains with three pores. The poppy capsule fragments are epidermis composed of small 5 to 6 sided cells with strongly thickened walls. Stellate lumina,
anomcytic stomata approximately 17 mm wide by 25 mm long or circular, are also infrequently noted.

**COLOR TESTS:**
As opium contains a mixture of various compounds, color test results may vary based on the components present and their concentrations.

**INSTRUMENTAL ANALYSIS:**
Common components of opium that may be encountered during instrumental analysis are codeine, noscapine, thebaine, morphine, and papaverine. At least four (4) common components of opium must be conclusively identified to report opium on the Forensic Laboratory Report. The Seized Drugs Unit requires that the following three (3) components must be conclusively identified:
- Codeine
- Thebaine
- Morphine

And then at least one of the following:
- Noscapine
- Papaverine
3.7 Title: ANALYSIS OF FENTANYL AND OTHER FENTANYL-RELATED SUBSTANCES

If an item is suspected to contain fentanyl or another fentanyl-related substance, the following procedures must be followed:

1. Wear proper PPE
2. Use proper engineering controls
3. Another person shall be in the Chemistry Laboratory and have access to Narcan.
4. Gross weight will be measured and documented in the case notes. If the item is confirmed to not contain fentanyl or another fentanyl-related substance then a net weight may be measured and documented in the case notes.
5. Sampling of the item will be done in a way to minimize aerosolizing the suspected fentanyl or fentanyl-related substance.
6. Samples that are suspected to contain fentanyl or fentanyl related substances should not be completely removed from their packaging. A small portion of the sample will be removed, weighed, analyzed, and repackaged separately into a laboratory bag. Homogeneity will only be ensured for the small portion that was removed for analysis. The weight of the entire submission along with the weight of the removed, weighed, and analyzed portion will be reported using sample selection wording.
7. At a minimum, the item must be packaged and individually sealed into three (3) layers of packaging. The outermost bag must be a nylon bag that is heat sealed. To minimize aerosolizing the substance, the heat sealer used will not evacuate the air from the bag that it is sealing.
8. The package containing the items should be placed into an airtight metal can or plastic bucket, if available..
9. Write “Fentanyl” on the outside of the package near the evidence label.
The clandestine laboratory response team responds to scenes where clandestine laboratory (clan lab) evidence needs to be sampled and/or collected. Each responding chemist is required to attend an initial 40-hour Hazardous Waste Operations and Emergency Response (HAZWOPER) or equivalent course to fulfill OSHA requirements. Every year, each responding chemist on the team is required to pass the 8-hour clandestine lab refresher course, complete fit-testing with their mask(s) and complete a medical examination provided by LVMPD.

Typically at a clandestine lab scene, the relevant chemicals/glassware should be brought to a well-ventilated area. Crime Scene Analysts may take photographs and process for latent prints, after which the responding chemist may collect samples. Photographs after sampling should also be taken either by a Crime Scene Analyst and/or Narcotics officers. A contracted hazardous waste company should be called by the case officer to dispose of the remaining items after sampling.

Clandestine laboratory samples collected in the field may be booked by the Forensic Scientist and then stored in the Chemistry Detail secured evidence storage room refrigerator. Notify an Evidence Technician of the booked clandestine laboratory samples as soon as possible after collection so that the samples may be data entered into ACE. Prior to analysis, clandestine laboratory evidence will be internally moved from the fridge location, via ACE, to the assigned Forensic Scientist.

**SAFETY CONSIDERATIONS:**
Care should be exercised when responding to clandestine labs. In many cases, samples come from containers that may be mislabeled. The samples are frequently very toxic, corrosive or flammable, and sometimes highly reactive. The responding chemist should obtain as much information about the clan lab as possible in order to be aware of the hazards to which they may be exposed.

**RESPONDING TO A CALL FOR SERVICE:**

- If dispatch calls the clan lab cell phone, ask for the case officer/designee’s information (name and contact number), then contact him/her and gather information about the potential clandestine laboratory. Information to gather and consider may include but is not limited to:
  - Case officer’s name and contact number if he/she contacted the clan lab phone directly
  - If not a Narcotics detective, has Narcotics been notified
  - The location of the scene
  - Do they have a search warrant
The primary event assigned to the operation
- What is present at the scene
- What type of manufacturing lab the officer suspects is present
- What types of samples they want the Chemist to collect
- The number of items that could be sampled
- Whether a hazardous waste cleanup company will be called

- Inform the case officer/designee that the Forensic Laboratory’s primary purpose at the scene is to collect samples only, and verify that an approved contracted hazardous waste cleanup company will be contacted to properly dispose of the remaining chemicals. The case officer/designee must also notify LVMPD Narcotics about the potential clan lab scene. It is preferred that an LVMPD Narcotics detective be on scene.

- Contact the Chemistry Laboratory Manager/designee for authorization to respond.

- In regards to outside agency requests for clandestine laboratory response, the LVMPD Department Manual Section 5/209.02 states that “requests made by other law enforcement agencies to perform crime scene processing may require the notification or approval of Executive Staff.” If an LVMPD event number is NOT assigned to a case and no LVMPD Narcotics officers are present at the scene, then the Chemistry Laboratory Manager/Laboratory Director must be notified before responding.

- When authorization is received, notify the requesting officer, collect all gear (e.g. masks, tanks, suits, safety goggles, gloves, sampling containers, sampling kit, notepad, pens/markers, etc.), load the clandestine laboratory vehicle and proceed to the clandestine lab scene.

- LVMPD Dispatch should be notified of in-service time, arrival time, cleared time and secured time.

  Notified: Time the initial call was received  
  In-Service: Time you started heading to the scene  
  Arrived: Time you arrived at the scene  
  Cleared: Time you are leaving the scene  
  Secured: Time you arrived back at the Forensic Laboratory

  NOTE: All traffic laws must be observed at all times and all passengers’ seat belts must be properly fastened.

Safety
- Wear appropriate personal protective equipment (PPE) at the scene. The level of protection may be dictated by the on-scene Site Safety Officer.

Photography
- Photographs should be taken by a Crime Scene Analyst and/or Narcotics officers at the scene.
• It is ideal to have photographs taken of the lab related items in place before removal or handling by a chemist.

• Photographs taken by the Crime Scene Analyst and/or Narcotics officers should include the original container from which a sample was taken by a chemist.

• Photographs of the samples should have the event number, analyst’s impound item number, initials of the chemist and the date visible.

**Items/Sampling**

• All sample packaging should include the event number, analyst’s impound item number, date and chemist’s initials.

• For liquid samples, a separate sample should be taken from each layer of a multi-layer item.

• If a liquid sample will be taken, the pH should be determined at the scene. The pH should be documented in the impound notes.

• For liquid samples, at least 30 mL or the entire sample will be recovered at the scene, whichever volume is less.

• Estimated volumes of other items that are **not** sampled, but may be used in the clandestine process may be documented in the impound notes. This will be at the discretion of the chemist at the scene.

• Items that contain solid material at the scene should be weighed on a balance located on the clandestine lab vehicle, prior to sampling. This weight must be documented in the impound notes. Finished product may be collected by the chemist but shall be booked by the officer.

• The balance used by the chemist must be checked using at least one of the weights on the clandestine laboratory vehicle and must be documented in the impound notes (CS# and mass of weight).

**Impound Notes**

The notes that are collected by the chemist will be considered impound notes. All drafts of the impound notes will be scanned and added to the unit record Object Repository assigned to the clan lab response in LIMS. Originals will then be shredded after verifying that all pages are properly uploaded in LIMS.

The first page of the impound notes shall include:

• Event number
• Date
• Case Officer
• Call out time
• Location
• Responding chemist name or P#/initials
Each additional page of the impound notes after page one shall include:
- Event number
- Responding chemist initials
- Agency (if not LVMPD)
- Corresponding page number
- Lab case # after entering the event into LIMS

The notes should also include:
- Brief description of each item sampled including color, state of matter (liquid, solid, etc.)
- Estimated quantity at scene (volume and weights)
- Results of preliminary field test performed by responding chemist
- Estimated applicable tablet count (include mg per tablet) from boxes or blister packs at scene
- pH of liquids
- Solvents, reagents and other lab related items that are not sampled or impounded by the Forensic Scientist

**Booking of Evidence**
Samples should be placed in overpack containers prior to booking. All samples will be packaged in such a way as to prevent loss, contamination or deleterious change during transport. At the earliest possible time, properly place the over packed samples into an appropriate cardboard box and place a blue "Refrigerator" sticker on the box. Properly label and book the samples through the evidence desk at the Forensic Laboratory. After the samples have been logged into ACE, place the package(s) into the appropriate location (i.e. refrigerator) for storage. If a request for analysis is not submitted within six (6) months of the scene response date, send the evidence to the Evidence Vault for long term storage.

**Guidelines for Sampling at Clandestine Labs**
- All sampling should be done in a well-ventilated area.
- A sample of any precursor chemicals should be obtained.
- Sampling of reagents and solvents is usually not necessary, but is at the discretion of the chemist.
- Refer to current local statutes to determine type and amount of sample needed
- Labeled containers whose contents are not consistent with the label should be sampled. It is common for suspects to repackage chemicals, reaction mixtures and/or waste.
- Factory sealed containers typically do not need to be sampled. There are exceptions to this rule and sampling will be at the discretion of the chemist.
- Any suspected reaction mixtures should be sampled.
- Each layer of a multi-layer solution should be sampled separately.
- Liquid samples are placed in a glass vial with a Teflon lined cap which should then be placed into an overpack container.
Solid samples (filters, powder samples, etc) may be sampled in the same manner as liquids or placed into a plastic bag. Glass vials are ideal since static electricity on plastic bags can create difficulties when analyzing the samples. Be aware that some acidic solid samples may deteriorate the plastic bag and glass vials will need to be used.

- Used glassware and equipment should be considered contaminated and treated appropriately. Do not book used glassware or chemical containers. Ideally, wait until CSAs have processed for prints before handling glassware/containers at the scene.

- Restock the clandestine lab vehicle with supplies after returning to the Forensic Laboratory. This may be done the following business day.

Safe handling, use, transportation and storage of measuring equipment

Manufacturer’s Operating or Instruction Manual(s) should be referred to when there are concerns about the handling, usage, transportation and storage of the clandestine laboratory balances.

Specific balances used for clandestine laboratory response remain on the response vehicle when not in use at a scene. These balances will be stored in their assigned carrying case during transport and when not in use.

When a balance is in use, place on a hard level surface. Perform a QC check on each balance prior to use. Use the appropriate weights located inside the carrying case to perform this QC check. Document the results of the QC check in the clandestine laboratory response notes.

Glassware is not typically transported out of the Forensic Laboratory, but may be needed on rare occasions for clandestine laboratory response. If glassware is used for measuring clandestine laboratory samples, it will be handled with appropriate personal protective equipment. It will be clean and inspected prior to use to be free of cracks and/or chips. If glassware is deemed unsuitable for use, it will not be used and will be disposed of in an appropriate receptacle at the scene.

Any glassware used at a clandestine laboratory scene shall be properly disposed of at the scene.
The objective in analyzing clandestine laboratory (clan lab) evidence is to determine if a purported clan lab site has the capacity to manufacture an illicit substance or in fact has already done so.

Samples usually contain a variety of liquids, solids, pure reagents, precursors, reaction mixtures, extracts and waste chemicals. These samples may be organic or inorganic. Solutions may be aqueous, organic, acidic or basic. Not all samples may need to be analyzed if finished products, controlled substances in solution, or precursors are found to be present. Reaction mixtures or waste with traces of controlled substances and other substances may be encountered.

Production reports will not be issued, and extrapolations of weights/volumes will not be calculated.

SAFETY CONSIDERATIONS:
Care should be exercised in analyzing samples from clan labs. In many cases, samples come from containers which may be mislabeled. The samples are frequently very toxic, corrosive or flammable, and sometimes highly reactive.

INTERPRETATION OF RESULTS:
In order to interpret the results of the analysis in this type of case, one must be familiar with at least one of the more common synthesis methods and procedures for the manufacturing of street drugs. The identity of precursors, reagents, finished products, and by-products must be considered as a whole to determine the intent of the clandestine chemist.

It is best to familiarize oneself with the suspected synthesis route(s) prior to analyzing the samples. This will provide the analyst with knowledge of potential precursors, intermediates and products that may be identified during their analysis. Other drug synthesis routes may also provide useful information during the analysis of samples.

The analyst should be familiar with household chemicals and how they can be used by the clandestine chemist, as well as what their legitimate use(s) may be. Sometimes nothing more than acids, strong bases and a few organic solvents are discovered. Often many of the reagents found are common household chemicals such as drain cleaner, painting solvents or battery acid.
ANALYTICAL ROUTE:
It should be noted that the nature of the sample determines the analytical route the analyst pursues and that many samples do not lend themselves to an exact order of analysis. Ultimately, the analyst decides the specific analytical route for each sample. The samples analyzed from a clan lab will be broadly categorized as either liquids or solids.

- When analyzing samples from a clan lab, the pH of the sample is not critical but it should be basic (pH ≥ 10) and is most commonly adjusted using NaOH. Sodium bicarbonate is not desirable due to the evolution of CO$_2$. NH$_4$OH is acceptable; however, it may add an unnecessary amount of liquid to the extraction tube.

- Petroleum ether and hexanes are most commonly used for cleaning up unwanted by-products in clan lab samples (pH acidic) but may also be used for isolating neutrals, methamphetamine and amphetamine (pH basic). These will typically form the top layer of a two layer liquid extraction.

- Chloroform and methylene chloride are most commonly used in isolating the compounds of interest from a clan lab sample (pH basic); however, they are heavier than water and will typically settle as the lower layer of a two layer liquid extraction.

- Pseudoephedrine and ephedrine must be derivatized in order to differentiate one from the other when using the GC/MS. Derivatizing is also helpful when attempting to identify amines which are present in very low concentrations or in unique matrices such as used red phosphorus. A 1% propyl chloroformate (PCF) solution is a derivatizing agent that reacts with phenethylamines in a variety of sample types. 1% PCF may be used as the organic extraction solvent after the pH adjustment of the sample and will typically form the top layer of the two layer liquid extraction.

Analyzing Liquid Samples

Characterize the liquid

- Calculating the density (D) of the liquid may be useful in characterizing the sample.

In general:
- D = 0.95 - 1.0 aqueous
- D = 0.80 - 0.90 miscible in organic (acetone, MeOH)
- D = <0.80 immiscible in organic (Coleman fuel, mineral spirits)
- D = >1.0 concentrated acids or bases

- Determine if the liquid is aqueous or organic.

- Determine if the liquid is miscible with an organic solvent.
If the analyst characterizes the liquid as a possible iodine solution/tincture, the liquid should first be analyzed as an unknown liquid utilizing the protocols below. The analyst may then attempt to precipitate solid iodine from the liquid and analyze the solid/crystals as outlined in the section titled Analyzing Powders/Solids, Iodine.

**Extract the liquid**
The method of extracting a liquid sample is solely dependent upon what was revealed in the characterization of the liquid.

### Aqueous

- Determine the pH of the sample

- **If the pH is basic:** A basic pH most commonly suggests a pH adjusted reaction solution or wash solution. It may contain residual amounts of a controlled substance and by-products and may demonstrate manufacturing of a controlled substance.
  - Place approximately 0.5 mL of the liquid into a test tube
  - An acid wash may be performed (optional)
  - If the pH is not already >10, add NaOH, vortex
  - Add equal volume of organic solvent, vortex, centrifuge
  - Remove the organic layer for analysis by GC/MS

- **If the pH is acidic:** An acidic pH most commonly suggests a reaction mixture solution that may contain a controlled substance and may demonstrate manufacturing of a controlled substance.
  - Place approximately 0.5 mL of the liquid into a test tube
  - Add an equal volume of hexanes (optional), vortex, centrifuge
  - Retain acidic (lower) layer, add NaOH solution until basic (pH >10), vortex
  - Add equal volume of organic solvent, vortex, centrifuge
  - Remove the organic layer for analysis by GC/MS

### Organic

- Determining the pH of an organic liquid is not necessary but may be accomplished by first moistening the pH test strip with deionized/distilled water.

- **If the organic liquid is miscible with H₂O:** A miscible organic solution most commonly suggests a tablet extraction solution (MeOH, etc.) and may demonstrate intent to manufacture.

  **Option 1:**
  - Place approximately 0.5 mL of the liquid into a test tube
  - Add an equal volume of deionized/distilled water
  - Add NaOH (pH>10), vortex
  - Add equal volume of organic solvent, derivatizing agent, vortex, centrifuge
  - Remove organic layer for analysis by GC/MS
Option 2:
- If the sample is miscible in water and it is organic, the liquid is most likely an alcohol
- Perform a Chen’s color test on the liquid
- Take the liquid sample and do a base to chloroform extraction
- Place the chloroform layer in a mortar/watch glass and bubble concentrated hydrochloric acid through liquid
- Dry down the liquid and perform an IR on the residue
- If the IR spectrum shows contamination, perform a GC/MS on a base to chloroform extraction

- **If the organic liquid is immiscible with H₂O:** An immiscible organic solution most commonly suggests a solvent used to extract a controlled substance from the reaction mixture such as Coleman fuel, mineral spirits, etc. Compounds of interest may be extracted from the liquid by performing the following:
  - Place approximately 0.5 mL of the liquid into a test tube
  - Add an equal volume of 0.2N H₂SO₄ or 2N HCl, vortex, centrifuge
  - Add an equal volume of hexanes (optional), vortex, centrifuge
  - Retain acidic layer, add NaOH (pH>10), vortex
  - Add equal volume of organic solvent, vortex, centrifuge
  - Remove organic layer for analysis by GC/MS

**Analyzing Powders/Solids**

**Extraction options for miscellaneous powders/red phosphorus**

- **Option 1**
  - Add small quantity of powder to test tube
  - Add approximately 1 mL deionized/distilled water
  - Add NaOH solution (pH>10), vortex
  - Add equal volume of organic solvent, vortex, centrifuge
  - Remove the organic layer for analysis by GC/MS

- **Option 2** (not appropriate for red phosphorus)
  - Add small quantity of powder to a vial
  - Dilute with 10% diethylamine in hexanes or appropriate solvent, vortex
  - Analyze by GC/MS

- **Option 3**
  - Add 200 mg of powder to filter column
  - Wash filter column with 2-3 mL of acetone, collect wash which may contain neutrals, such as, nicotinamide or dimethylsulfone.
  - To acetone wash, add 10 mL of petroleum ether to precipitate the nicotinamide or dimethylsulfone, swirl, filter, dry, perform infrared analysis
  - Wash filter column with 2-3 mL of chloroform, collect wash which may contain a controlled substance
The following protocol applies to solid forms of iodine such as elemental iodine crystals, prill, or solid residues; however, the procedure for precipitating solid iodine from an iodine solution/tincture is also noted. Iodine solutions or tinctures can be analyzed as an unknown liquid utilizing the protocols outlined in the section titled Analyzing Liquid Samples or with instrumental analysis to identify iodine. The Forensic Scientist may then attempt to precipitate iodine crystals from the tincture solution for analysis.

**Iodine Precipitation**
- Place approximately 1 mL of the solution/tincture into a test tube
- Add a few drops of HCl, vortex (optional)
- Add approximately 1 mL of 3% hydrogen peroxide
- Iodine crystals will precipitate out of solution, filter
- Perform following tests on solid crystals
- Note: This test will not work with povidone iodine solutions

**Solubility test**
- Add small quantity of solid into test tube
- Add approximately 1 mL of chloroform
- A violet color is indicative of iodine
- Add an equal volume of methanol
- A brown color indicates the presence of iodine

**Vapor/Crystal test**
- Add small quantity of solid to test tube, cap
- Heat the tube in a heat block or over a flame in the chemical fume hood
- Watch for formation of violet vapors
- Remove from heat and allow to cool
- Iodine crystals will form upon cooling
- View with stereoscope, iodine crystals appear as bluish-black squares/plates/feathery like needles with a metallic luster

**Color test**
- Add small quantity of solid to a spot plate well, test tube or filter paper
- Use spray starch solution,
- A bluish purple color will develop in the presence of iodine
- Quality Control:
- **Positive:** Iodine, Bluish-purple
- **Negative:** Empty well/test tube/ filter paper, No color change
**Note:** If attempting to detect the presence of iodine in a solid sample such as suspected used red phosphorus or a suspected reaction mixture, wash the sample with deionized water and perform the color test on the wash solution as noted above. If a bluish/purple color does not develop, add a drop of 3% hydrogen peroxide, a subsequent development of a bluish/purple color indicates the presence of iodine.

**OR**

- **Add** a small quantity of solid or stained filter to test tube containing methanol
- **Separate** methanol solution into two (2) test tubes,
  - In one (1) test tube add 5% silver nitrate - formation of yellow precipitate is indication of presence of iodine.
    - **Quality Control:**
      - **Positive:** Iodine in MeOH  Yellow precipitate
      - **Negative:** MeOH  No color change
  - In the other test tube use spray starch - formation of bluish/purple color is indication of presence of iodine.
    - **Quality Control:**
      - **Positive:** Iodine in MeOH  Bluish/purple
      - **Negative:** MeOH  No color change

- **Instrumental Analysis of solid or liquid iodine via XRF**
  - **Add** enough sample to cover the bottom of a clean XRF sample cup and cover with appropriate plastic film
  - If there is not enough sample to cover the bottom, then use entire sample for analysis with the XRF
  - Analyze by XRF using the appropriate method

- **Instrumental Analysis of liquid iodine via GC/MS**
  - **Add** a small quantity of sample to a culture tube
  - **Add** an equal volume of deionized/distilled water and a few drops of NaOH
  - **Add** chloroform to extract iodine, vortex and centrifuge
  - **Transfer** chloroform layer to a GC/MS vial for analysis on the instrument
  - Identification of triiodomethane indicates presence of iodine in sample

- **Instrumental Analysis of solid iodine via GC/MS**
  - **Add** a small quantity of sample to a culture tube
  - **Add** absolute ethanol and let soak for a couple of minutes
  - **Add** an equal volume of deionized water and a few drops of NaOH
  - Let all the iodine dissolve, or transfer the aqueous liquid into another culture tube before proceeding to the next step
  - **Add** chloroform to extract iodine, vortex and centrifuge
  - **Transfer** chloroform layer to a GC/MS vial for analysis on the
Phosphorus

**Precipitation/Color test**
- Add small amount of suspected red phosphorus to test tube
- Add 0.5 mL deionized/distilled water, vortex, centrifuge
- Acidify washed sample with 3 - 4 drops of $\text{HNO}_3$
- Add 3-4 drops of ammonium molybdate solution $[(\text{NH}_4)_2\text{MoO}_4]$, vortex
- Gently heat to almost boiling for ~ 2 minutes
- Formation of a finely divided yellow precipitate confirms the presence of the phosphate ion

**Quality Control:**
- **Positive:** Red Phosphorus    Yellow precipitate
- **Negative:** Empty test tube    No precipitate

**Microscopic examination I**
- Prepare a wet mount of the suspected red phosphorus using glycerol, a microscope slide and a cover slip.
- View under a microscope, red phosphorus appears as amorphous crystals resembling red stained glass

**Microscopic examination II**
- Place a small amount of sample into a test tube.
- Follow the procedure below in a separate clean test tube to prepare a negative control.
- In the hood, add a few drops of concentrated nitric acid. Enough acid should be added to make the sample wet or slightly liquid. A few drops of deionized/distilled water may be added to non-powder samples (coffee filters) in order to prevent over-drying. Coffee filters may also be rinsed with deionized/distilled water, and the rinsate placed into the test tube as the coffee filters may burn. Upon addition of the acid, orange fumes and effervescence may occur.
- Slowly heat the test tube in a flame and allow the wet sample to boil.
- Keep heating/boiling the sample while occasionally pulling the test tube out of the flame to check if the orange fumes have subsided.
- When the orange fumes have subsided, place a drop of the solution onto one half of a glass slide.
- Place a drop of saturated ammonium molybdate on the other half of the glass slide.
- Using the end of a clean glass pipette or similar instrument, draw the two drops together and streak the mixture across the
middle of the slide. Scratching the slide with the pipette may be necessary to initiate the crystallization.
  - Wait for a slight yellow-green color to appear on slide (or wait 1-2 minutes).
  - View slide utilizing a microscope with transmitted light.
    Phosphate crystals will appear as yellow-green, 4-pointed, 3-dimensional stars.

Quality Control:
  - Positive: Red phosphorus
  - Phosphate crystals
  - Negative: Blank reagents
  - No phosphate crystals

- **Instrumental Analysis of red phosphorus via XRF**
  - Add enough sample to cover the bottom of a clean XRF sample cup and cover with appropriate plastic film
  - If there is not enough sample to cover the bottom, then use entire sample for analysis with the XRF
  - Analyze by XRF using the appropriate method

**CAUTION:** The following procedure converts red phosphorus to white phosphorus. Due to the hazardous nature of white phosphorus, the heating of the sample with the propane torch must be completed in the fume hood and the sample must be capped. White phosphorus can spontaneously ignite in the presence of air; however, it is stable in chloroform.

- **Instrumental Analysis of white phosphorus via GC/MS**
  - Add approximately 2-3 mg of suspected red phosphorus to test tube and push on cap
  - In the fume hood, using a test tube holder heat the test tube with a propane torch until sample starts to smoke
  - Allow the test tube to cool inside the fume hood.
  - In the fume hood, add approximately 1 mL of chloroform to the test tube and cap
  - Gently agitate the sample
  - Analyze by GC/MS using the appropriate method.
Seized Drug laboratory reports will be in declaration format.

All items received in the package(s) by the analyst will be included on the laboratory report, including those items that were received, but not examined. Items listed on the laboratory request, but not received by the analyst do not need to appear on the report.

If a sample is examined using only literature reference source comparison criteria, the report shall state “This determination was made from a comparison of the physical characteristics of the above listed commercially manufactured preparation(s) to a literature reference source(s). Conclusive testing must be completed prior to trial.” This statement will suffice as the method of testing for associated items.

If a sampling plan used, it shall be documented on the laboratory report. The following statement must appear on the report when the hypergeometric statistical sampling plan is used: “A hypergeometric statistical sampling plan was used to determine the number of samples to be tested. This plan provides a 95% confidence level that at least 90% of the samples contain the substance(s) reported.”

If an item is examined using sample selection, the official laboratory report shall state that limited analysis was performed, e.g. “Analysis was only performed on a limited sampling of Lab Item ## listed above. Further testing may be required prior to trial.”

The total number of packages containing the items received shall also be reported, e.g. “Four (4) packages containing numerous tablets”.

“No controlled substance(s) and/or dangerous drug(s) were conclusively identified” shall be used in cases that do not meet the conclusive identification criteria. “No controlled substance(s), dangerous drug(s), and/or precursor chemical(s) were conclusively identified” shall be used in clandestine laboratory analyses. In certain circumstances, it may be necessary for the analyst to report more detailed results than described above.

The category A techniques that were used to analyze each item will also be included on the laboratory report. Mass spectrometry will be reported as a hyphenated technique with gas chromatography, e.g. “Lab Item 1 was tested using Gas Chromatography-Mass Spectrometry (GC-MS). Lab Item 2 was tested using Fourier Transform Infrared Spectroscopy (FTIR).”

Items reported as marijuana will list all methods of testing.
Examples of the instrumental methods that may be included on the laboratory report are:
- Fourier Transform Infrared Spectroscopy (FTIR)
- Fourier Transform Infrared Spectroscopy-Attenuated Total Reflectance (FTIR-ATR)
- Gas Chromatography-Mass Spectrometry (GC-MS)
- Raman Microscopy
- X-ray Fluorescence (XRF)

The following statement must be included on the report when weight(s) are being reported: “A coverage probability of approximately 95% was utilized in the calculation of uncertainty (+/-) for the measurement(s) reported above.”

Non-controlled substances will be footnoted with the phrase, “not a controlled substance.” Non-controlled substances that require a prescription will be footnoted with the phrase, “not a controlled substance, prescription required.”

The start and end dates of testing will appear on the laboratory report. The testing and sampling statements will appear as:
“That the start date of testing is <date> and the end date of testing is <date>; That sampling took place between the start and end dates of testing;”
Dates should be in numerical format and must include the month, day and year.

The start date of testing is when the LIMS worksheet is created. If LIMS is not functional, then the start date of testing will be when the analysis began and will be documented on the LVMPD ISD 27/ISD 27a Controlled Substances Worksheet. The end date of testing will be the date that the latest report (i.e. the report that will be released) was generated.

Lab Number is titled “Lab Case Number” on the report. Lab Number and Lab Case Number are synonymous.

The following table lists wording to be used on the report for cases involving the listed scenarios:

<table>
<thead>
<tr>
<th>Scenario</th>
<th>Report Wording</th>
</tr>
</thead>
<tbody>
<tr>
<td>Syringes accompanied by other seized drug evidence</td>
<td>“Due to their potential hazards, syringes are not routinely analyzed” will be reported under Results, Opinions, and Interpretations</td>
</tr>
<tr>
<td>Phenacetin is conclusively identified</td>
<td>List as a footnote: “Banned in the US”</td>
</tr>
<tr>
<td>Federally exempted prescription products that appear on the current Exempt Prescription Products List published by the DEA Diversion Control Division</td>
<td>List as a footer: “Item # is listed as a Federally exempt prescription preparation”</td>
</tr>
<tr>
<td>Methorphan Propanoxyphene MDEA</td>
<td>List as a footnote: “Isomer not determined”</td>
</tr>
</tbody>
</table>
### Form of amisole

“PTHIT” will be reported under Results, Opinions, and Interpretations. The PTHIT will be footnoted with: “Phenyltetrahydroimidazothiazole, not a controlled substance”

### Reanalysis of an item(s)

“Lab Item(s) ## were previously examined by <Analyst name> P#<#####>. See Forensic Laboratory Report of Examination with distribution date mm/dd/yyyy.”

This statement must appear on the report above the “I declare…” statement.

### Missing item

Description column will state “Not received” and the Results, Opinions, and Interpretations column will state “Not examined”

---

Any diagrams, sketches or photographs used to depict the location of sampling are considered notes. If they are deemed necessary for the interpretation of the results either by the analyst or through the review process, they must be included on the formal laboratory report.
LVMPD FORENSIC LABORATORY
TECHNICAL PROCEDURES
SEIZED DRUGS

5.2 Title: TECHNICAL REVIEW

The purpose of technical review is to confirm that the conclusions of the analysts are supported by the data and documented results of the analyses. Technical reviews are to be conducted by a Forensic Scientist II or Chemistry Laboratory Manager who has expertise in the specific seized drugs discipline (e.g. clandestine lab response and analysis, general unknowns, etc.) and has been proficiency tested in that area. Analysts are responsible for checking their own queue on a daily basis to see which cases are awaiting their review.

All casework will be subjected to technical review.

Technical reviews will be performed by reviewing all data, notes and reports associated with the case. A technical review is not an independent verification and should not typically involve reanalyzing the raw data, verifying details of library entries, checking previous version histories of worksheets, or other aspects that are more typical of an audit trail than a case file review.

A Seized Drugs technical review worksheet/checklist will be completed by the technical reviewer in LIMS. The technical review will be undertaken as soon as practical after the case is completed.

If the report is cancelled in LIMS because the case was returned to the analyst during an administrative review, LIMS automatically assigns a new technical review. This technical review must be completed, unless the administrative review was returned for any of the following reasons:
- Billing activity was not completed
- Items in the Resource Manager need to be added or removed (without editing the worksheet)
- Spelling and grammar that does not affect the results, opinions, or interpretations
- Minor punctuation edits that do not alter the meaning of the sentence or phrase

If any of the above criteria applies, the additional technical review can be withdrawn in LIMS.

When the electronic or paper version of form ISD 27/ISD 27a is used, each case will be stamped “Technical Review by:__________________” (sic) in the upper right corner of the first page of case notes. The technical reviewer of the case will write their initials and P# on the blank line of the stamp during the technical review process, and will use the Seized Drugs Technical Review Form located in Qualtrax.
Retraining of analysts may be required under the following four (4) circumstances:

Failed qualitative proficiency test

Failed clandestine laboratory scene response proficiency test

Problems identified during casework or during supervised casework

Long period of absence (>6 months)

Short absences (<6 months) will require the analyst to review all Department and Forensic Laboratory manual changes, and all Chemistry Detail meeting minutes that were recorded during their absence. If deemed necessary by the Chemistry Laboratory Manager a condensed competency test may be given and must be successfully completed prior to casework.

Seized drugs analysis is very dynamic, making it nearly impossible to create hard and steady rules for making identifications applicable to every scenario. For example, is it acceptable to report NCSD if an antibiotic was present but not detected, even though the Seized Drugs Technical Manual was followed? The answer to this question is not straightforward, and will depend on the circumstances surrounding the particular case (e.g. what form was the evidence received in, were other tests performed, what instrumentation was used, were there any other indicators, etc.). Therefore, the ultimate decision on which individual situations require retraining will be made by the Chemistry Laboratory Manager, in conjunction with the Quality Manager. The details pertinent to each individual case (e.g. was the Seized Drugs Technical Manual followed, did the testing procedure follow a logical train of thought, was there a shortcoming in the laboratory’s policy, etc.) will be evaluated and documented.

Failed qualitative proficiency test:
Any incorrect/missed identifications of qualitative controlled substance(s)/dangerous drug(s) will be evaluated and reviewed in order to determine the root cause. The appropriate corrective action will be determined by the Chemistry Laboratory Manager, in conjunction with the Quality Manager. If the analyst is required to repeat the qualitative analysis and all the drug(s) of interest are identified correctly during the repeat analysis, the results will be deemed satisfactory. Failure to identify the drug(s) of interest a second time will warrant further investigation/action by Laboratory Management. The determined root cause will direct whether or not the analyst may continue with casework.

The identification and/or reporting of adulterants/diluents/sugars is not mandatory.
Clandestine laboratory proficiency test results will be evaluated on a case by case basis. The ability of the LVMPD Forensic Laboratory to properly analyze these samples may be limited due to the capabilities of the instrumentation.

**Failed clandestine laboratory response proficiency test:**
Clandestine laboratory response proficiency tests will be created internally. If an analyst fails the proficiency test, the deficient areas will be evaluated by the Chemistry Laboratory Manager. The extent of retraining will be decided by the Chemistry Laboratory Manager in conjunction with the Quality Manager.

**Problems identified during casework or during supervised casework:**
Any problems identified during supervised casework or independent casework/reviews will be brought to the attention of the Chemistry Manager. The extent of retraining will be decided by the Chemistry Laboratory Manager in conjunction with the Quality Manager.

**Long periods of absence (>6 months):**
In instances where an analyst has been out of the Seized Drugs Unit (e.g. on leave, transferred, TDY, etc.) for an extended period of time (>6 months), completion of a competency test will be required. A short re-training period may be necessary prior to completion of the competency test. The extent of retraining will be decided by the Chemistry Laboratory Manager in conjunction with the Quality Manager.
COLOR TEST RECIPES
The following pages contain recipes and quality control information for color test reagents used in the Seized Drugs Unit.
AMMONIUM MOLYBDATE

Use: This reagent is used to aid in the preliminary identification of red phosphorus.

Limitations:
Both phosphates and arsenates react with ammonium molybdate to form insoluble yellow precipitates, therefore arsenates must be removed before the confirmatory test for phosphates can be made. Presence of arsenates may be determined with XRF analysis.

Materials:
- Ammonium Hydroxide
- Molybdic Acid
- Nitric Acid
- Deionized/Distilled Water

Reagent Preparation:
Dissolve 5 grams of molybdic acid in a mixture of 15 mL deionized/distilled water and 7.5 mL of concentrated ammonium hydroxide. Add this solution slowly and with constant stirring to a mixture of 57 mL of deionized/distilled water and 25 mL of concentrated nitric acid.

OR

To make a saturated solution of ammonium molybdate in H$_2$O
At room temperature, 1 gram of ammonium molybdate dissolves in approximately 2.3 mL H$_2$O.

OR

Add solid ammonium molybdate to sample directly in the presence of nitric acid

Testing Instructions:
Add a small amount of red phosphorus in a test tube.
Acidify with nitric acid.
Add 3-4 drops of ammonium molybdate solution and heat. Formation of a yellow precipitate indicates the presence of the phosphate ion.
NOTE: Precipitation takes place more readily at ~60°C in the presence of excess nitric acid.

Quality Control:
Positive: Red Phosphorus Yellow ppt. (With heat)
Negative: Empty test tube No color change

Storage: One time use

Disposal:
Spot Test – dispose into acid waste
Bottle – pour into an appropriately sized amber bottle with screw cap, ensure the cap is tight, and place into the acid waste container in the hazardous waste storage room
CHEN’S REAGENT

Use: This reagent is used to aid in the preliminary identification of ephedrine and pseudoephedrine.

Limitations:

Not specific for any one substance.
Limited sensitivity.
The addition of reagents are order dependent.

Materials:

Glacial Acetic Acid
Cupric Sulphate
Sodium Hydroxide
Deionized/Distilled Water

Reagent Preparation:

Reagent 1) For a 1% acetic acid solution, mix 0.5 mL of glacial acetic acid into 49.5 mL of deionized/distilled water.
Reagent 2) For a 1% cupric sulphate solution, dissolve 0.5 gram of cupric sulphate into 50 mL of deionized/distilled water.
Reagent 3) For a 2 N sodium hydroxide solution, dissolve 4 grams of sodium hydroxide into 50 mL of deionized/distilled water.

Testing Instructions:

Add small amount of sample to a spot plate well.
Add one or two drops of Reagent 1, then one or two drops of Reagent 2, and one or two drops of Reagent 3. Observe any color change within 1 minute.

Quality Control:

Blank: Blue
Positive: Pseudoephedrine Violet
Negative: Caffeine No color change

Storage: Room temperature

Disposal:

Spot Test – dispose into base waste
Reagent 1 – pour slowly into acid waste
Reagent 2 – pour slowly into acid waste
Reagent 3 – pour slowly into base waste
1% COBALT NITRATE

Use: This reagent is used as a screening aid for gamma hydroxybutyric acid.

Limitations:

Not specific for any one controlled substance.
Limited sensitivity.
This test does not work very well with liquid samples.
KOH and NaOH will give a gray color.

Materials:

Cobaltous Nitrate
Methanol

Reagent Preparation:

Dissolve 3.9 g of cobaltous nitrate in 500 mL of methanol.

Testing Instructions:

Add small amount of sample to a spot plate well.
Add one or two drops of reagent and observe any color change.

Quality Control:

Positive: GHB Pale purple
Negative: GBL No color change

Storage: One time use

Disposal:

Spot Test – dispose into corrosive acid waste
Bottle – pour into an appropriately sized amber bottle with screw cap, ensure the cap is tight, and place into the corrosive acid waste
COBALT THIOCYANATE REAGENT

Use: This reagent is primarily used to aid in the preliminary identification of cocaine.

Limitations:

Not specific for any one substance.
Limited sensitivity.

Materials:

- Cobalt Thiocyanate
- Deionized/Distilled Water
- 2N HCl

Reagent Preparation:

Add 2 grams of cobalt thiocyanate to 100 mL deionized/distilled water.
Stir to dissolve.
Filter

Testing Instructions:

Add small amount of sample to a spot plate well.
Add one or two drops of reagent and observe any color change.
For suspected cocaine base, add one or two drops of 2N HCl.

Quality Control:

Positive: Cocaine HCl Blue
Negative: Caffeine No color change

Storage: Room temperature

Disposal:

Spot Test – dispose into base waste
Spot Test with 2N HCl – dispose into acid waste
Bottle – pour slowly into base waste
TOLUENE/COBALT THIOCYANATE

Use: This reagent is used as a screening aid for gamma butyrolactone.

Limitations:

Not specific for any one controlled substance.
Limited sensitivity.
A blue color will develop with ether and ethanol solutions.

Materials:

Toluene
Solid Cobalt Thiocyanate crystals

Sample Preparation:

Add approximately 1 mL of sample into a test tube followed by 0.5 mL of toluene.
Vortex.

Testing Instructions:

Place a few crystals of solid cobalt thiocyanate in a ceramic spot well and add
3.5 drops of the toluene layer.
Observe for a color change upon drying.
Running a blank is suggested.

Quality Control:

Positive: GBL Blue
Negative: GHB No color change

Storage: One time use

Disposal:

Spot Test – rinse insolvent waste
Bottle – pour slowly into solvent waste
DILLE-KOPPANYI REAGENT

Use: This reagent is used to aid in preliminary identification of barbiturates.

Limitations:
Not specific for any one substance.
Limited sensitivity.

Materials:
Reagent 1) 0.1% solution of cobaltous acetate in a 0.2% solution of glacial acetic acid in methanol

- Cobaltous Acetate 0.05 g
- Glacial Acetic Acid 0.1 mL
- Absolute Methanol 50.0 mL

Reagent 2) 5% solution of isopropylamine in absolute methanol

- Isopropylamine 2.5 mL
- Absolute Methanol 47.5 mL

Reagent Preparation:
Reagent 1) Deliver cobaltous acetate to a 100 mL flask or beaker. Add glacial acetic acid. Swirl to dissolve. Add absolute methanol. Swirl to mix.

Reagent 2) Combine isopropylamine and absolute methanol in a beaker. Swirl to mix.

Testing Instructions:
Add small amount of sample to a test tube. Add one or two drops of reagent 1. Add one or two drops of reagent 2 and observe for color change.

Quality Control:
Blank: Pink
Positive: Phenobarbital Lavender
Negative: Caffeine No color change

Storage: Refrigerator

Disposal:
Reagent 1 – dispose in corrosive acid/flammable waste
Reagent 2 – dispose in solvent waste
Spot Test – dispose into corrosive acid waste
Bottle – pour slowly into corrosive acid/flammable waste
MODIFIED DUQUENOIS LEVINE

Use: This reagent is used to aid in the identification of marijuana.

Limitations:

- Not specific for marijuana without additional testing.
- Limited sensitivity.
- The addition of reagents are order dependent.
- The stability of the color reaction is time dependent.
- Immature, old or burnt marijuana may not yield strong color results.

Materials:

- 200 Proof Ethyl Alcohol/95% Ethyl Alcohol
- Deionized/Distilled water
- Acetaldehyde
- Vanillin
- Concentrated HCl
- Chloroform

Reagent Preparation:

- Prepare 200 mL of a 95% solution of ethyl alcohol by adding 10 mL of deionized/distilled water to 190 mL of 200 proof ethyl alcohol.

- Add 1 mL of Acetaldehyde and 4 g of the Vanillin to the 200 mL 95% Ethyl Alcohol (see above)

- Concentrated Hydrochloric Acid
- Chloroform

Testing Instructions:

1. Place a small amount of suspect material in a test tube.
2. Cover the material with Reagent 1 (Duquenois Levine). Mix gently.
3. Add an equal amount of Reagent 2. Mix gently and observe for a color change in this aqueous layer.
4. Add enough Reagent 3 to visualize an appropriate separation. Mix gently and observe for an extracted color in the lower organic layer.

Quality Control:

- Blank: Straw over Colorless
- Positive: Marijuana Blue-Purple (aqueous) over Purple (organic)
- Negative: Tobacco Straw over Straw
- Negative: Coffee grounds Reddish-Brown (aqueous) over Light Pink (organic)
Stability/Storage:

Store in an amber glass stoppered bottle and inside chemical fume hood.
Discard if reagent turns yellow.

Disposal:

Color Test – dispose into corrosive acids/flammable waste
Reagent 1 – pour slowly into solvent waste
Reagent 2 – pour slowly into corrosive acid waste
Reagent 3 – pour slowly into solvent waste
5% FERRIC CHLORIDE

Use: This reagent is used to aid in the preliminary identification of gamma-hydroxybutyric acid.

Limitations:
- Will react to any alkaline solution.
- Limited sensitivity.

Materials:
- Ferric Chloride
- Deionized/Distilled Water

Reagent Preparation:
Dissolve 5 g of ferric chloride in 100 mL of deionized/distilled water.

Testing Instructions:
Add small amount of sample to a spot plate well.
Add one or two drops of the reagent. Observe any color change within 1 minute.

Quality Control:
- Positive: GHB                Reddish-orange precipitate
- Negative: GBL               No color change

Storage: Refrigerator

Disposal:
Spot Test – dispose into corrosive acid waste
Bottle – pour slowly into corrosive acid waste
FROEHDE REAGENT

Use:  This reagent is used to differentiate opiate compounds.

Limitations:

- Not specific for any one substance.
- Limited sensitivity.

Materials:

- Molybdic Acid
- Concentrated Sulfuric Acid

Reagent Preparation:

Add 0.1 gram molybdic acid to 20 mL concentrated sulfuric acid. Heat with constant stirring to dissolve.

Testing Procedure:

Add small amount of sample to a spot plate well. Add one or two drops of reagent and observe any color change.

Quality Control:

<table>
<thead>
<tr>
<th>Positive</th>
<th>Caffeine</th>
<th>No color change</th>
</tr>
</thead>
<tbody>
<tr>
<td>Heroin</td>
<td>Purple</td>
<td></td>
</tr>
</tbody>
</table>

Storage:  Room Temperature

Disposal:

- Spot Test – dispose into corrosive acid waste
- Bottle – pour slowly into corrosive acid waste
LIEBERMANN REAGENT

Use: This reagent is used as a general screening test.

Limitations:

- Not specific for any one substance.
- Limited sensitivity.

Materials:

- Potassium Nitrite
- Concentrated Sulfuric Acid

Reagent Preparation:

Add 10 gram potassium nitrite, VERY CAREFULLY, in small amounts to 100 mL concentrated sulfuric acid.
Swirl between additions.
Divide into dropper bottles.

Testing Instructions:

Add small amount of sample to a spot plate well.
Add one or two drops of reagent and observe any color change.

Quality Control:

Positive: Methadone Yellowish-Orange
Negative: Mannitol No color change

Storage: Room Temperature

Disposal:

Spot Test – dispose into corrosive acid waste
Bottle – place into Liebermann waste
MANDELIN REAGENT

**Use:** This reagent is used as a general screening test.

**Limitations:**

- Not specific for any one substance.
- Limited sensitivity.

**Materials:**

- Ammonium Vanadate
- Deionized/Distilled Water
- Concentrated Sulfuric Acid

**Reagent Preparation:**

- Dissolve 0.5 gram ammonium vanadate in 1.5 mL deionized/distilled water, heat if necessary.
- Bring to 100 mL with concentrated sulfuric acid, then filter with glass wool.
- Divide into dropper bottles.

**Testing Instructions:**

- Add small amount of sample to a spot plate well.
- Add one or two drops of reagent and observe any color change.

**Quality Control:**

- **Positive:** Methadone Greenish-blue
- **Negative:** Caffeine No color change

**Storage:** One time use

**Disposal:**

- Spot Test – dispose into corrosive acid waste
- Bottle – pour slowly into corrosive acid waste
MARQUIS REAGENT

Use: This reagent is used as a general screening test.

Limitations:

Not specific for any one controlled substance.
Limited sensitivity.

Materials:

37% Formaldehyde Solution
Concentrated Sulfuric Acid

Reagent Preparation:
Add 1.0 mL of 37% formaldehyde solution to 100 mL concentrated sulfuric acid.
Mix. Divide into dropper bottles.

Testing Instructions:
Add small amount of sample to a spot plate well.
Add one or two drops of reagent and observe any color change.

Quality Control:

<table>
<thead>
<tr>
<th>Positive</th>
<th>Methamphetamine</th>
<th>Orange to Brown</th>
</tr>
</thead>
<tbody>
<tr>
<td>Positive</td>
<td>Heroin</td>
<td>Purple</td>
</tr>
<tr>
<td>Negative</td>
<td>Caffeine</td>
<td>No color change</td>
</tr>
</tbody>
</table>

Storage: Room temperature

Disposal:
Spot Test – dispose into corrosive acid waste
Bottle – pour slowly into corrosive acids/flammable waste
MAYER REAGENT

Use: This reagent is used to aid in preliminary identification of phencyclidine (PCP) and other alkaloid drugs.

Limitations:

- Not specific for any one substance.
- Limited sensitivity.

Materials:

- Hydrochloric Acid
- Mercuric Chloride
- Potassium Iodide
- Deionized/Distilled Water

Reagent Preparation:

- Reagent 1) For a 2N HCl solution, add 5 mL of concentrated hydrochloric acid into 30 mL of deionized/distilled water.
- Reagent 2) Dissolve 0.5 gram mercuric chloride into 50 mL of deionized/distilled water. Slowly add potassium iodide and an orange precipitate will form. Continue adding the potassium iodide until precipitate dissolves.

Testing Instructions:

Place a small amount of sample onto a dark colored spot plate. Add two drops of Reagent 1 and then add a few drops of Reagent 2. Observe for a precipitate.

Quality Control:

- Blank: No precipitate
- Positive: PCP White precipitate
- Negative: Caffeine No precipitate

Storage: Chemical fume hood

Disposal:

- Spot Test – dispose into corrosive acid waste
- Reagent 1 – pour slowly into corrosive acid waste
- Reagent 2 – pour into an appropriately sized amber bottle with screw cap, ensure the cap is tight, and place into the acid waste
MECKE REAGENT

Use: This reagent is used as an aid in identification of opiate compounds.

Limitations:

Not specific for any one substance.
Limited sensitivity.

Materials:

Selenous Acid
Concentrated Sulfuric Acid

Reagent Preparation:

Add 0.25 gram selenous acid to 25 mL concentrated sulfuric acid.
Swirl to dissolve.

Testing Instructions:

Add small amount of sample to a spot plate well.
Add one or two drops of reagent and observe any color change.

Quality Control:

Positive: Heroin Blue-Green
Negative: Caffeine No color change

Storage: Room temperature

Disposal:

Spot Test – dispose into corrosive acid waste
Bottle – pour slowly into corrosive acid waste
SODIUM NITROPRUSSIDE TEST

Use: This reagent is used to help in the identification of compounds having a secondary aliphatic amine (-CH₂-NHCH₂). This test is useful in distinguishing amphetamine, a primary amine, from methamphetamine, a secondary amine.

Limitations:
Not specific for any one substance.
Some secondary amines may not react because of steric hindrance.

Materials:
- Sodium Nitroferricyanide
- Deionized/Distilled Water
- Acetaldehyde
- Sodium Carbonate

Reagent Preparation:
Reagent 1) 1% solution of sodium nitroferricyanide:
Add 1.0 g sodium nitroferricyanide to 90.0 mL of deionized/distilled water. Mix to dissolve sodium nitroferricyanide. Then, add 10.0 mL of acetaldehyde to solution and mix.

Reagent 2) 2% sodium carbonate:
Add 2.0 g of sodium carbonate to 100 mL of deionized/distilled water and mix.

Testing Instructions:
Add small amount of sample to a spot plate well.
Add one or two drops of nitroprusside reagent.
Add one or two drops of sodium carbonate reagent and observe any color changes.

Quality Control:
Blank: Pinkish-Orange
Positive: Methamphetamine Blue
Negative: Pseudoephedrine No color change

Stability/Storage: Light and heat sensitive, store at room temperature

Disposal:
Spot Test – dispose into corrosive base waste
Reagent 1 and 2 – pour slowly into corrosive base waste
VAN URK REAGENT

Use: This reagent is used to aid in the preliminary identification of LSD and psilocin.

Limitations:

- Not specific for any one substance.
- Limited sensitivity.
- If LSD blotter paper is colored, color reaction may be difficult to interpret. A methanol extraction may be necessary before testing can begin.

Materials:

- 1% solution of p-dimethylaminobenzaldehyde in a 1:1 solution of MeOH and HCl

  p-Dimethylaminobenzaldehyde 0.25 g
  Hydrochloric acid 12.5 mL
  Methanol 12.5 mL

Reagent Preparation:

- Mix equal parts of the hydrochloric acid and methanol.
- Add p-dimethylaminobenzaldehyde to mixture.
- Stir to dissolve.

Testing Instructions:

- Add small amount of sample to a spot plate well.
- Add one or two drops of reagent and observe any color change.

  OR

- Dissolve sample in MeOH and place dropwise onto filter paper with a capillary tube.
- Add one or two drops of reagent and observe any color change.

Quality Control:

- Positive: LSD Light Purple
- Negative: Caffeine No color change

Storage: Refrigerator

Disposal:

- Spot Test – dispose into corrosive acid/flammable waste
- Bottle – pour slowly into corrosive acid/flammable waste
WEBER TEST

Use:

This reagent is used to aid in the preliminary identification of psilocin (found in mushrooms).

Limitations:

Not specific for any one substance.
Will react with phenolic groups.

Materials:

Fast Blue B or Fast Blue salt BN (solid)
Deionized/Distilled Water
Concentrated Hydrochloric Acid

Reagent Preparations:

Reagent 1) Add 0.1 g of Fast Blue B or Fast Blue salt BN to 100.0 mL of deionized/distilled water. Stir to dissolve.

Reagent 2) Hydrochloric Acid

Testing Instructions:

In a test tube, add a small quantity of mushroom.

Add enough of Reagent 1 to cover the mushroom. Shake/agitate the above mixture for approximately one minute or until a color change is observed.

Next, add a couple of drops of hydrochloric acid to the above mixture and observe any color change.

Quality Control:

Positive: Psilocin Red upon addition of Reagent 1, and Blue upon addition of Reagent 2.

Negative: Culinary Mushrooms No reaction.

Storage: One time use

Disposal:

Spot Test – dispose into corrosive acid waste
Reagent 1 – pour slowly into corrosive base waste
Reagent 2 – pour slowly into corrosive acid waste
DERIVATIZING AGENTS
The following lists specific derivatizing agents used in seized drugs analyses:

BSTFA + 1% TMCS - (N,O-bis [Trimethylsilyl]trifluoroacetamide + Trimethylchlorosilane)

MSTFA - (N-methyl-N-trimethysilyltrifluoroacetamide)

TPC – N-trifluoracetyl-L-prolylchloride

1% PCF - Propyl chloroformate (see preparation instructions below)

**Use:**
This is used to derivatize amines.

**Materials:**
Propyl Chloroformate
Hexanes
Chloroform

**Preparation:**
To prepare 10 mL of a 1% PCF solution (in 3:1 hexanes:chloroform), place 7.5 mL of hexanes into a 10 mL graduated cylinder. Add 2.5 mL of chloroform. Pipette 100 uL of PCF into cylinder. Transfer solution to labeled and capped storage bottle. Swirl to mix.

**Stability/Storage:**
Stable for one year at room temperature.

**Disposal:**
Dispose into solvent waste

OTHER COMMON RECIPES

**Acidified Methanol:**
1 drop concentrated hydrochloric acid
20 drops methanol
Expires: 1 year (or earliest expiring component)
**Storage:** Room temperature

**2.0 N Ammonium Hydroxide (Starting with a 14.8 N solution) (7.4 mL):**
1mL ammonium hydroxide
6.4 mL deionized/distilled water
Expires: 1 year (or earliest expiring component)
**Storage:** Room temperature

**2N Hydrochloric Acid (HCl) (30 mL):**
5 mL concentrated hydrochloric acid
Volume to 30 mL with deionized/distilled water  
Expires: 1 year (or earliest expiring component)  
**Storage:** Room temperature  

### 1% PCF Derivatizing Agent (202 mL):

- 150 mL hexanes  
- 50 mL chloroform  
- 2 mL PCF (propylchloroformate)  
Expires: 1 year (or earliest expiring component)  
**Storage:** Room temperature  

### Saturated Sodium Bicarbonate (NaHCO$_3$) (250 mL):

- 17.50 g sodium bicarbonate  
- 250 mL deionized/distilled water  
Expires: 1 year (or earliest expiring component)  
**Storage:** Room temperature  

### ~5% Silanizing Agent (210 mL)

- 200 mL toluene  
- 10 mL chlorotrimethylsilane or hexamethyldisilazane  
Expires: One time use  
**Storage:** One time use  

### 5% Silver Nitrate (10 mL)

- 0.5 gram silver nitrate  
- 10 mL deionized/distilled water  
Expires: One time use  
**Storage:** One time use  

### 2% Sodium Carbonate (Na$_2$CO$_3$) (1 L):

- 20 g sodium carbonate  
- 1 L deionized/distilled water  
Expires: 1 year (or earliest expiring component)  
**Storage:** Room temperature  

### 1 N Sodium Hydroxide (NaOH) (1 L):

- 40 g sodium hydroxide  
- 1 L deionized/distilled water  
Expires: 1 year (or earliest expiring component)  
**Storage:** Room temperature  

### 50% Sodium Hydroxide (NaOH) (500 mL):

- 250 g sodium hydroxide  
- 500 mL deionized/distilled water  
Expires: 1 year (or earliest expiring component)  
**Storage:** Room temperature  

### 0.2 N Sulfuric Acid (H$_2$SO$_4$) (180 mL):

- 1 mL concentrated sulfuric acid  
- 179 mL of deionized/distilled water
Expires: 1 year (or earliest expiring component)
**Storage:** Room temperature

### 4:1 Petroleum Ether:Diethyl Ether (Volume varies)
Combine four parts petroleum ether with one part diethyl ether.
Expires: Until consumed (or earliest expiring component)
**Storage:** Room temperature

### Fast Blue 2B Spray (100 mL):
Combine 0.1g Fast Blue 2B salt and 70 mL of absolute ethanol. Bring to a volume of 100 mL with water.
Expires: Until consumed (or earliest expiring component)
**Storage:** Refrigerator

The course of seized drugs analysis cannot always be anticipated. Preparation of other reagents may be required depending on the nature of the analysis. These instances will be documented in the Resource Manager or case record (if one time use), but may not necessarily be listed in the Seized Drugs Technical Manual.

**Recipes for VIPER and Auto Theft**
No storage conditions are listed, because these chemicals are made at the time VIPER requests them and are not stored in the Forensic Laboratory.

### Heyn's Solution (24 mL):
- Cupric ammonium chloride: 1 g
- Concentrated Hydrochloric acid: 12 mL
- Deionized water: 12 mL
**Filter solution**
Expires: Until consumed

### 5% Sodium Hydroxide (NaOH) (100 mL):
- Sodium hydroxide: 5 g
- Deionized water: 100 mL
Expires: Until consumed
8.1 Title: LOGGING REFERENCE MATERIALS INTO LIMS

It is not necessary to complete each step in the order presented, but all steps need to be completed.

This procedure is for entering reference materials into the LIMS only and serves as a supplement to the “Verifying New Reference Materials” procedure. New reference materials received in the lab will also have to be entered into the Chemistry Inventory spreadsheet in Excel, and will need to be validated. Refer to the “Procedure for Verifying New Reference Materials” for instructions.

1. Obtain the Certificate of Analysis (C of A) and MSDS/SDS for the lot# either from the manufacturer’s website, or by contacting the manufacturer directly. If the laboratory is already in possession of the most current version of the MSDS/SDS, a second copy does not need to be obtained. You will need electronic copies of the C of A and the MSDS/SDS. Refer to the “Procedure for Verifying New Reference Materials” for directions on where to save the electronic copies.


3. Find the name of the reference material. If you cannot find it, look for any synonyms or acronyms (e.g. look for GHB when looking for gamma hydroxybutyric acid). If you still cannot find it, request that the Resource Manager Admin for the Chemistry Detail create a folder for it. Give him/her the name of the compound and let him/her know if it is a primary or secondary reference material so that it can be added to the appropriate folder.

4. Once the folder has been created, highlight it.

5. On the top left of the screen, select “New.” The Resource Details window will open.

6. At a minimum, you must enter in the lot#, Date in Service, Location in which the reference material will be stored, Asset ID (which is the manufacturer), Manufacturer, and Description.
   a. The description field should contain the name of the substance with the lot# in brackets (e.g. Heroin [12345-6 A]).
   b. In the Comments section, add the expiration or retest date, if one is listed on the bottle or on the relevant paperwork. If there is no expiration or retest date, indicate in the Comments section that the expiration date is until consumed (e.g. EXP: U/C). For all reference materials (with the
exception of reference materials that were purchased at, and are intended to be used at a known concentration), 9/9/9999 should be entered into the Expiration Date field. For reference materials that were purchased at, and are intended to be used at, a known concentration, enter in the manufacturer’s expiration date into the Expiration Date field. 

c. The terminology used for the Location field should be consistent with the terminology in the Chemical Inventory spreadsheet, preceded by “CHEM – ” (e.g. CHEM – Lower Flammables Cabinet).

7. The expiration/re-test date can be found on the packing slip, the bottle, the C of A, or may be indicated on an Expiration Date Letter created by the manufacturer. The Expiration Date Letters are stored in H:\CB\Forensics\General\Chemical Inventory (MSDS)\Expiration Date Letters.

8. Save and Close. Re-open the resource. Click on “Manage Files” from the ribbon.

9. Select Import from the ribbon. Choose the MSDS/SDS and the C of A for that reference material. Select Open, make any changes to the name of the file, then Save and Close. The name of the file should indicate what the file is (e.g. Heroin C of A, Heroin MSDS, etc.) Double click the files you just imported to ensure that they are the correct documents. If they are, close the documents, highlight them from the list, and select Approve from the ribbon. If they are not the correct files, highlight them from the list and select Delete. Begin again to upload the correct files. Close the Object Repository window, and Save and Close the Resource Instance Details. Once the C of A and MSDS/SDS have been imported into the Object Repository for that resource, they can be deleted from your computer. If the MSDS/SDS has a newer revision date than one that the laboratory already had on file, email (or print a hard copy and give) this to the LEST in charge of filing MSDS/SDS’s. The MSDS/SDS can now be deleted from the temporary location in which it was saved.

10. Any other optional notes can be added to the Comments section on the first “General” tab of the Resource Details window (e.g. scheduling status, synonyms, acronyms, physical state, etc.).

11. Double check that all dates are consistent, all entries are complete, and all information is accurate. Save and Close.
8.2 Title: VERIFYING REFERENCE MATERIALS

It is not necessary to complete each step in the order presented, but all steps need to be completed.

All reference materials will be verified upon receipt, prior to being placed in permanent storage. A complete verification includes obtaining structural data either by using the GC/MS and/or the IR, uploading the C of A and SDS into LIMS, and following the steps below regarding labeling, library entry, and logging into the laboratory’s electronic systems. GC/MS data will include at least one Helium spectrum and one Hydrogen spectrum (when obtainable). IR data will be obtained when the reference material cannot be differentiated from its isomers using the GC/MS, when the isomer distinction has a state or federal statutory effect, or when the hydrogen spectrum does not match an approved library source and a helium spectrum is not obtainable. The Chemistry Manager may also require that IR data be obtained in other circumstances (e.g. pseudoephedrine vs. ephedrine, etc.).

1. For newly received reference materials, write the received date, and your p#/initials on the new reference material bottle. If the substance received was ordered using a DEA 222 form, retrieve the retained copy of the form from the Chemistry Manager’s office and write the quantity and date received in the appropriate fields. See older copies for an example if unsure of where to write the information. For in-house reference materials, ensure that a received date is on the bottle with someone’s p# and initials. If it is not there, alert the Chemistry Manager. Some research through the lab’s archived files may need to be performed.

2. Obtain electronic copies of the Certificate of Analysis (C of A) and the most current version of the MSDS/SDS from the manufacturer/vendor’s website. Save these on your Desktop, or other temporary location from which you can easily retrieve them.

3. Write the expiration date on the reference material bottle, if it is not there already. You can also use a sticker label. In the event that an expiration date is not indicated by the manufacturer, check the folder located at H:\CB\Forensics\General\Chemical Inventory (MSDS)\Expiration Date Letters to see if a declaration from the manufacturer has been obtained in regards to the stability of the product. You can also find the expiration date on the Certificate of Analysis, packing slip or by contacting the manufacturer directly. Unless the reference material was ordered at, and will be used at, a known concentration (e.g. column test check purchased at 1mg/mL), the reference material will be on a “retest prior to use” status. The expiration date must still be on the bottle, but
the laboratory will not discard the reference material once this date has passed. For reference materials that were purchased at, and will be used at, a known concentration, the manufacturer's expiration date must be honored.

4. Upload the MSDS to the Controlled Substances MSDS folder (H:\CB\Forensics\General\Chemical Inventory (MSDS)\Chemistry\Seized Drugs) and name the MSDS by reference material name and manufacturer (e.g. “Codeine Sigma”). It is only necessary to add/update an MSDS if one does not exist already or a new version is available. You may have to log onto the manufacturer's website or call them to obtain a copy of the SDS for that lot#. Give a hard copy of the SDS to the person assigned to that duty so that he/she can file the SDS in the appropriate binders in the bullpen and send a copy to the Safety Detail of the Department.

5. Make a new entry alphabetically for the reference material lot# in the “All Drugs” tab in the Chemistry Inventory spreadsheet. Complete all columns across the row. Make a separate entry for every bottle/vial received in the Excel Inventory, the Drug Weight History sheet, and in LIMS.
   a. If multiple bottles were received with the same lot#, add a letter designator at the end of each lot# (e.g. Heroin lot# 123456-7 A, Heroin lot# 123456-7 B, etc.).
   b. If the reference material is a controlled substance, add a column for that lot# to the “Drug Weight History” sheet in Qualtrax, following the format of the current entries. Make a new column for every bottle/vial received. Each bottle/vial must be weighed separately, but QC checking one bottle will suffice for every bottle/vial with the same manufacturer lot# (see step 6 for weighing procedures).
   c. If the new reference material is also a secondary reference material, check the “Chemistry Secondaries” tab for the most recent lot# to assign to the new reference material. Write the lot# and name in the “Chemistry Secondaries” tab and proceed with the entry in the “All Drugs” tab and “Drug Weight History” sheet. Also remember to add the secondary lot# to the bottle/vial.

6. If the new reference material is a controlled substance, measure the initial gross weight using an analytical balance before opening/using the reference material. Record the initial gross weight in the “Drug Weight History” sheet using the same format as other entries already in the form. If the reference material is a secondary reference material, include the tablet/capsule etc. count on the “All Drugs” tab. If the secondary reference material is also controlled, add the tablet/capsule count to the “Drug Weight History” sheet as well.

7. Make a new entry for, or find, the reference material lot# in the LIMS Resource Manager. Refer to the “Procedure for Logging Reference Materials into LIMS” for instructions.

8. If you haven’t done so already, write the open date and your p#/Initials on the reference material bottle.
9. Prepare a master stock solution vial of the reference material to be stored in CS freezer #5. Create aliquot(s) of this stock solution and analyze the reference material on at least one GC/MS instrument using helium as a carrier gas (if possible), and one GC/MS instrument using hydrogen as a carrier gas. The FTIR instrument may also be used if necessary as per the preface to this procedure. Compare all spectra to an approved published literature source.

10. Review the data. If the data is acceptable and if the reference material spectrum needs to be added to the library for any instrument, attach an approved, published literature spectrum or a spectrum from another approved, published library. Note that not all instrument libraries can be used for verification (Cayman, etc.). Stamp the first page of the file with the MS library stamp and complete all fields (e.g. chemical formula, molecular weight, CAS #, msclip range, library used). Give the data to the Chemistry Manager/designee to approve, along with the data from the published literature source. If any spectra are not acceptable, re-analyze the reference material. You may have to use extraction techniques in order to obtain acceptable data.

11. If the reference material spectrum does not need to be added to the library, you may use an LVMPD library to verify the reference material as long as the manufacturer and lot# of the library entry and the manufacturer and lot# of the new reference material are not the same. Upload the analysis data into LIMS, approve the files, and skip to step 13.

12. After the data has been approved for library entry and has been returned to you, complete steps 12.a through 12.d. If the reference material was analyzed on the FTIR, follow the Procedure for Adding Spectra to the FTIR/ATR Library, located in the Chemistry Training Program folder in Qualtrax. If the reference material has already been added to the mass spectrum libraries, skip to step 15. **The reference material spectrum can only be added to the library for the instrument on which it was analyzed.**

   a. Perform the MS clip (if needed) using the same range that you included on the data when requesting approval from the Manager/designee
   b. Add the mass spectrum to the library through the Data Analysis software by going to Spectrum→Edit Library→Select Library. Navigate to the LVMPD library for that instrument and select it. Click OK. Now select Add New Entry, click OK.
      i. Add the compound name, the molecular formula, and the CAS # in the designated fields, and add the manufacturer and lot # in the miscellaneous information field. Be sure that the “Include in Search” button is selected. Click OK, click OK, then click Cancel. To find the library entry number, run a search on the mass spectrum so that it matches to itself. Write down the library entry number on the data.
   c. Copy the electronic raw data file(s) that were entered into the library and paste into the library folder for that particular instrument. The data file path may vary based on the instrument being used. Once the file(s) is in the library folder, rename the file to be the name of the reference material, remembering to keep the “.D” file path extension (e.g. Methamphetamine.D).
d. Scan the data with the library entry number written on it to the appropriate library folder.

13. Record the retention time(s) in the appropriate Retention Time form for that instrument. If retention time data already exists for that reference material (on that instrument and method), add the new manufacturer/lot#/retention time/library entry number/date for that reference material, preferably on a new line in the same cell. Complete this information whenever the retention time has shifted. Save the data file in the appropriate electronic Retention Time folder.

14. Repeat steps 12-13 for all other applicable GC/MS instruments on which the reference material was analyzed.

15. Scan and import the packing slip, product information sheet(s), spectrum from one helium (if obtainable) and one hydrogen instrument, data from the FTIR (ATR), published literature spectrum, and/or any other relevant documentation that was received with the reference materials into the resource details for the reference material in LIMS. Double click on the file(s) to make sure the whole file and correct information was imported, then close and click approve. Once the imported completed packet has been verified by the analyst, the original hard copy can be put in a shred basket. Write “QC Checked” and your P#/initials on the reference material bottle and place it in the proper storage location, per the manufacturer’s guidelines, or in your appropriate box to be weighed back in by another analyst if it is a controlled substance.

16. If the material cannot be QC checked for whatever reason (e.g. doesn’t chromatograph, no acceptable literature source to compare, etc.), place it in a plastic bag, write “QC Check needed” along with the name of the material on the outside of the bag, and place it in the appropriate storage location per the manufacturer’s recommendations.
LVMPD FORENSIC LABORATORY
TECHNICAL PROCEDURES
SEIZED DRUGS

8.3 Title: LOGGING USED REFERENCE MATERIALS INTO THE INVENTORY

It is not necessary to complete each step in the order presented,
but all steps need to be completed.

The Laboratory’s controlled substances reference material collection may be
used by authorized personnel for casework comparisons, training, research,
quality control checks, and various other tasks. When controlled reference
materials are used by any authorized personnel, the gross weight of the
reference material must be logged by a second authorized employee prior to
placing the reference material back in its storage location. Authorized personnel
include but may not be limited to Forensic Laboratory Aide, Forensic Scientist
Trainee, I, and II positions assigned to the Chemistry Detail, the Chemistry
Manager, other authorized Laboratory Managers, and all those in the Chemistry
Manager’s chain of command with access to the Drug Vault. For the purpose of
this procedure, authorized personnel will be referred to as “analyst” or
“personnel.”

1. When a controlled reference material has been used by an analyst, it will be
placed inside a box indicating that it needs to be logged into the inventory.
Boxes have been placed in various locations in the Drug Vault where personnel
can temporarily store their used reference materials until they are weighed and
placed back in the permanent storage location. The presence of a reference
material in one of these boxes indicates to all personnel that this reference
material requires weighing, logging, and placement back in permanent storage.

2. Boxes have been placed on the countertop in the Drug Vault, in the room
temperature desiccator, in CS Freezer #7, in CS Refrigerator #6, and in CS
Refrigerator #6 desiccator. Some boxes have been labeled with the name and/or
initials of the personnel who utilize the reference materials most often. If a box
has been labeled with your name/initia, place the controlled reference material
you used in this box. Use the box whose location most closely matches the
permanent storage location of your used reference material (e.g. if a reference
material belongs in the freezer desiccator, place it in the box in the freezer, etc.).

3. If a box has not been labeled with any name/initia, it should be labeled with a
phrase like “Needs Weighing,” or something similar, indicating the purpose of the
box. Place your used reference material in the box with a sticky note attached to
it. Write your name or p#/initials on the sticky note so that the second analyst
knows who used the reference material.

4. When placing used reference materials in the boxes, indicate (on a sticky note) if
you made any significant changes to the bottle that could affect the weight (e.g.
added a tape flag, repackaged due to broken container, etc.). If you used a large
quantity of the reference material (e.g. to re-make the reagent QC check vials, etc.), indicate (on a sticky note) what the reference material was used for. The analyst who weighs the reference material back in will need to know this information. The second analyst may approach you to ask for clarification. Any weight differences greater than ±5% will require a note explaining the difference, and the Chemistry Manager may need to be notified (see step 11 for more information).

5. All secondary reference materials will be counted after use. Controlled reference materials require a weight in addition to a count. The weight (and/or tablet/capsule count) of controlled secondary reference materials will be recorded in the Drug Weight History sheet. The counts of all controlled and non-controlled secondary reference materials will be recorded in the All Drugs tab of the Chemistry Inventory.

6. As a courtesy, it is helpful if an email is sent to the entire Chemistry Detail, or to the primary and secondary personnel assigned to this task, notifying them that reference material(s) need weighing. This will help ensure that the reference materials get weighed and replaced in the appropriate storage location in a timely manner so that others may use it if necessary. This also minimizes the amount of time that the reference material is outside of its permanent storage location.

7. A second analyst (one other than the analyst who used the reference material) must perform the following steps. Go to the Drug Vault and open the Chemistry Inventory spreadsheet located in the H:\ drive. Also check out the Drug Weight History spreadsheet from Qualtrax.

8. When weighing controlled reference materials back into the inventory, use the analytical balance in the Drug Vault whenever possible. Tare the balance between every weighing, and wait for the balance to steady at 0.00000 g prior to measuring the gross weight. If the analytical balance is not appropriate for the size of the container, use the (2) decimal place balance in the Drug Vault, or use one of the analysts’ casework balances at the Chemistry benches. One of the balances in the Drug Vault should be used whenever possible.

9. Most reference material weights are recorded to (2) decimal places only. Some reference material weights, however, are recorded to (4) decimal places. This is because the quantity of reference material is so small (1 mg, 5 mg, etc.), that recording the weight to (2) decimal places only records the weight of the container without taking into account the change in quantity inside the container. Most reference materials ordered from Cayman Chemicals have weights that are recorded to (4) decimal places. If you are unsure of how to record the weight, you may ask another analyst, or see how the reference material was weighed in previously. When in doubt, record to (4) decimal places.

10. When recording the weight on the Drug Weight History page, highlight the appropriate cell in the column for that reference material lot#. Enter in today’s date in the top row of the cell, and hit Alt+Enter to move the cursor to the next row within the cell. Enter in your p#/initials. In the next row of the cell, enter the gross weight that you measured, with units. In the next row of the cell, enter
in the name of the analyst who used the reference material. If there is an explainable discrepancy in the weight, make a note inside that cell (e.g. tape flag added to bottle; broken cap – sample repackaged, etc.). If the weight discrepancy exceeds ±5% and cannot be explained, notify the Chemistry Manager.

11. Open the “All Drugs” page in the Chemistry Inventory if the substance you weighed was a secondary reference material that consists of tablets, capsules, etc. Find the row for that reference material lot# that you just weighed/counted. Scroll right to the “Quantity on Hand” column and update the tablet/capsule etc. count.

12. Check the Chemistry Inventory for the location in which the reference material is to be stored. Double check that you are looking in the correct row, at the correct location. Place the reference material back in its permanent storage location. Save and close both spreadsheets. Check the Drug Weight History sheet back into Qualtrax and release it for approval.

13. If you notice inconsistencies or inaccurate information when you are working in the Chemistry Inventory for any reason, research what the correct information should be by comparing the reference material bottle, the Certificate of Analysis, the packing slip, the MSDS, and/or any other pertinent documents to each other. Make the corrections on all pertinent tabs in the Chemistry Inventory spreadsheet, LIMS Resource Manager, on the bottle, on the verification data, on the GCMS vial aliquot in CS Freezer #5, and/or any other relevant location.

14. No action needs to be taken in the LIMS Resource Manager regarding the weighing of controlled reference materials, unless no usable quantity is left and the bottle/vial is being disposed of. If no usable quantity is left, find the reference material lot# in the LIMS Resource Manager to Archive that lot#, and make the appropriate notation in the All Drugs page (see Procedure for Controlled Reference Material Audit for details on what the notation should include).
LVMPD FORENSIC LABORATORY
TECHNICAL PROCEDURES
SEIZED DRUGS

8.4 Title: MAKING REAGENTS

1. **Always** follow Good Laboratory Practice (GLP) and all safety procedures outlined in the Forensic Laboratory Quality Manual, the Forensic Laboratory Safety Manual, the Seized Drugs Technical Manual, and the Seized Drugs Training Manual.

2. Be sure you have a clean, uncluttered working space. Use of the lab aide fume hood is ideal for reagent preparation; however, certain other time sensitive circumstances or circumstances involving casework may take precedence. If this is the case, you may wait (for a short period of time), or move to another fume hood. Ensure that you have an uncluttered workspace by moving items to another fume hood. Be sure to replace the original items back into the appropriate fume hood when you are finished.

3. Read the recipe and the SDS for each chemical/material that you will use before starting. Get the most current recipe from the electronic version of the Technical Manual. Get the SDS from LIMS, the electronic file, or from the yellow folders in the bullpen. Be familiar with all the content in the SDS, but pay special attention to the hazards identification, fire and explosion data, accidental release measures, handling and storage, exposure controls/personal protection, physical and chemical properties, and stability and reactivity data sections.

4. Qualtrax contains the most updated version of laboratory forms. Retrieve the appropriate Reagent Prep form from Qualtrax. Most color test reagents should have their own specialized template. For a reagent/solution that does not require QC checks at regular intervals (with the exception of the Marquis reagent), use the General Reagent & Solution Prep Log.

5. All required chemicals/materials can be found in the refrigerator(s), chemical storage room, or Elga water system. Best practice is to carry one chemical at a time, and carry large bottles with one hand around the neck of the bottle and one palm supporting the bottom of the bottle. Use the rubber carriers located in the chemical storage room if necessary. **Never** carry any containers by the cap, or by the neck only. Always keep the door to the chemical storage room open if it is being occupied or used by a person.

6. Double check the name of the chemical(s) you retrieved and the name of the ingredient(s) listed in the recipe. The difference between potassium nitrite and potassium nitrate may only be one letter, but the chemical reactions can vary greatly when combining each one with a strong acids and chemicals.
7. Follow the recipe. Be sure to pay special attention to the specific order of processes, and certain key phrases like “add X to Y” as opposed to phrases like “mix X and Y.” For example, the recipe for the Duquenois-Levine reagent 1 directs you to add acetaldehyde and vanillin to ethanol. If you ignore the directional phrase and simply pipet the acetaldehyde into a beaker and then add the ethanol, the volatility of the acetaldehyde will likely cause it to evaporate before you add the ethanol, and your reagent will not pass the QC check. It is best practice that for volumes of 2 mL or less, an adjustable volume pipet is used, not a graduated cylinder. For volumes between 2 mL and 5 mL, it is best practice to use a 10 mL graduated cylinder (or adjustable volume pipet if you prefer). For quantities above 5 mL use an appropriately sized graduated cylinder. It is not ideal to measure volumes for a recipe in a glass beaker.

8. Be sure to dispose of all generated waste in the appropriate containers, per the Safety manual and SDS. Indicate which chemical(s) you are disposing of on the waste bucket sheets on the fume hood sash (for liquid waste only). When in doubt of where to dispose something, test its pH. Be sure to check the SDS for incompatibilities before disposing. Not all acids play well together, and some should be containerized separately prior to placing in the acid bucket. Be aware that some chemicals should have their own bucket entirely.

9. Assign an internal lot# to the reagent/solution that you are making. Sign in to Qualtrax and refer to the “Reagent Log Summary” form for the next available internal lot#. The form is located in the following file path: LVMPD → Forensic Lab → Chemistry → Reagent Prep and QC Sheets. When prompted, select “Open.” The internal lot# should begin with the letter ‘C’ and then consist a 6 digit number indicating the current month, day, and year, followed by a ‘-’ and a sequential number starting with 1. The sequential number will indicate the order in which reagents/solutions were made that day. For example, the first three reagents/solutions made on March 17th, 2014 would have the internal lot# structure as follows, C031714-1, C031714-2, C031714-3, etc. Assign the next sequential lot # for that day and enter it in the next empty cell in the Reagent Log Summary sheet and the Prep Log. Save the Reagent Log Summary sheet as a different name (any appropriate name) and close. Back in Qualtrax, select “Replace”. Browse for the file you just saved, select “Upload,” then select “Replace” again. When prompted, select “Open” to verify that the new version uploaded successfully.

10. Assign an expiration date based on the stability of the reagent/solution (monthly, annually, until consumed, etc.), or sooner based on the expiration date(s) of any components used to make the reagent/solution. The earliest expiration date should be used. Stability and expiration information can be found in the CS Technical Manual, or on the bottle from the old lot#. Check a calendar to make sure the expiration date does not fall on a weekend, a Friday, or a holiday. If it does, move the expiration date up to the last business day before the weekend/Friday/holiday. Enter the expiration date onto the Reagent Prep form.

11. Complete all appropriate fields in the preparation log and check the box labeled “Recipe and Initial QC Check”. If using water as part of the recipe, indicate whether the water is distilled/deionized (water from the Elga system is
12. Collect all the bottles of the old/current working lot#. Perform a QC check on the old/current lot# and the new batch that you just made in accordance with the directions in the recipe, if applicable. You will have to check out the working copy of QC checks for the current/old lot# from the Resource Manager in LIMS. Refer to the “Procedure for Reagent Quality Control Checks” for specific directions on performing QC checks, and appropriate actions in case a QC check fails. Document the results of each check on the appropriate preparation log, and mark “Final QC check” if applicable.

13. If all QC checks of the new lot# pass, aliquot the new reagent/solution into the appropriate number of containers. See the NOTE at the bottom of this procedure regarding which bottles to use. Use the pre-made reagent bottle labels located at H:\CB\Forensics\Chemistry\Reagents\Reagent Labels, or use the label maker (found in the top left drawer of the bench behind bench #5 by the windows) to label each container with the reagent/solution name, lot#, preparer’s p#/initials, and expiration date.

14. If not already on the label, place the appropriate GHS hazard label(s) on each bottle containing the newly prepared reagent/solution. These can be found in the green safety binder. Smaller labels may have to be taped on to prevent them from falling off. Also add a signal word sticker like, “Danger,” or “Warning.” These can also be found in the green safety binder. In addition, a label indicating the storage conditions must be placed on the container. These indicators are typically color coded (e.g. room temp, freezer, fridge, etc.).

15. During the initial preparation, all fields in the Reagent Prep Log should be completed. Ensure that this has been completed and save this file onto your desktop using the name of the reagent followed by the lot# in brackets and the phrase “Initial Preparation and QC Check” (e.g. Marquis [C111015-1] Initial Preparation and QC Check). Save the monthly and final QC Check form for the old lot # onto your desktop.

16. Follow the instructions in the “Procedure for Logging Reagents/Solutions Made In-House into LIMS” in the CS Technical Manual to enter the information of the new lot# into the LIMS.

17. Archive the old lot# in the LIMS Resource Manager so that no analyst continues to add this lot# to his/her worksheet. Open LIMS and go to the Resource Manager module → LVMPD Forensic Laboratory → Resources → Reagents/Solutions → Chemistry → Select the name of the reagent/solution that you are disposing of → double click the appropriate lot# to open the Resource Details window → Select the “Archived” button → Save and Close. The laboratory can only have one working lot# of a given reagent/solution at a time.

18. Open the Outlook shared Chemistry calendar. Find the last business day before the expiration date of the reagent/solution you just made. Reagents/solutions will be made the day before the expiration date to ensure that there is always a
working lot# available for use, and that there is only a minimal time period in which the reagent/solution is unavailable (during initial QC check and labeling prior to distribution). Select “New Appointment” from the ribbon. In the Subject line, enter “Make XXX” with XXX indicating the name the reagent/solution. Select “All Day Event.” Assign this appointment to the orange category. See below for the list of color categories.

19. In the Outlook shared Chemistry calendar, on the actual expiration date, select “New Appointment” from the ribbon. In the Subject line, enter “Exp: XXX” with XXX indicating the name the reagent/solution. Select “All Day Event.” Assign this appointment to the red category.

20. If this reagent/solution requires QC checks at regular intervals, go to the date of the next required QC check and create an appointment. In the Subject line, enter in “QC XXX” with XXX indicating the name of the reagent/solution. Select “All Day Event.” Assign this appointment to the purple category. See below for the list of color categories.

21. Go back to today’s date on the calendar. If there is an appointment on the calendar regarding the reagent/solution you just made, open the appointment by double clicking it and add the word “Complete” at the beginning of the Subject line. If you are making a reagent/solution prior to its expiration date because stocks were low, etc., find the actual expiration date of the reagent/solution in the Outlook calendar and delete the entry indicating that the lot # expires that day.

22. Distribute the container(s) of the new lot# (e.g. analysts’ benches, fume hood(s), etc.). Check the leftover quantities of the chemicals that you used from the chemical storage room/refrigerator, etc. Ensure that there is enough left for the next reagent/solution (of any type) that needs to be made. You can check the shared Chemistry calendar in Outlook to see what reagents are due to be made next. If quantities are low, order more. Return all chemicals that you used back to their appropriate storage locations (e.g. flammables cabinet, refrigerator, etc.). If you don’t remember where the chemicals go, check the Chemistry Inventory or the LIMS Resource Manager. If you consumed the entire quantity or there is not a usable quantity left, dispose of the chemicals in the appropriate waste container.

23. Dispose of the old lot# in the appropriate waste stream, and rinse all surfaces of all glassware, supplies and empty dropper bottles/caps from the old, disposed lot# thoroughly into the appropriate waste container, using an appropriate solvent for that chemical/waste container.
   a. The old dropper bottles/caps may require some vigorous scrubbing/wiping with a solvent soaked kimwipe/cotton swab/wooden stick to remove any staining. Be careful not to scratch the Teflon surface of the Teflon coated bottles/caps! You may soak the dropper bottles/caps in soapy water for a period of time, if you prefer. If staining still persists, try soaking the bottles/caps in a 3:1 solution of methanol : 1N NaOH, or a 3:1 solution of methanol : concentrated HCl. You can also sonicate the bottles/caps inside of this soak solution. If the staining cannot be
removed, discuss with the Chemistry Detail and Manager whether the bottles/caps should be discarded and replaced.

b. If the reagent was stored in an amber glass screw top bottle with green Teflon cap (e.g. Ferric Chloride, Van Urk, etc.), rinse the bottle thoroughly into the appropriate waste container and dispose of the empty bottle in the appropriate barrel/bucket in the chemical storage room.

c. If the reagent/solution was stored in a glass bottle with a plastic or glass stopper (e.g. Duquenois Levine, diluted acid, etc.), rinse the bottle and stopper thoroughly into the appropriate waste container, remove all labeling, and place the bottle and stopper in one of the wash basins. You may also wash and rinse the glass bottle/stopper and re-use it for the new lot# once it is completely dry. Note that residual water or acetone may affect the performance of the reagent/solution.

24. Place any re-usable, rinsed bottles and caps in the wash basins. Never put any dirty dishes directly into the sinks. If disposing of the lot# for any reason other than the approaching expiration date (e.g. low quantity necessitates the preparation of a new lot#), a note must be added to the comments section of the Resource Details window in LIMS as to why the lot# is being archived (e.g. consumed, failed QC check, etc.), along with the analyst's p#/initials and date.

25. When you are done, empty the dry waste buckets from the fume hood into the blue barrels in the chemical storage room. Mark the list of chemicals on the blue barrel in the chemical storage room accordingly. If the liquid waste container in the fume hood is full, cap it, write the end use date on the label, place it in the appropriate blue barrel in the chemical storage room and mark the chemical list accordingly by comparing the list from the hood sash to the list on the blue barrel (remove the sheet from the hood sash and take it with you). After verifying that all the appropriate chemicals have been checked on the list on the blue barrel, dispose of the sheet from the hood sash in the trash.

a. Create a new liquid waste bottle by grabbing an empty 1 L bottle (or 4 L if 1 L bottles are not available) from the cabinet in the chemical storage room. On a blank piece of half paper (pre-cut paper should be in the same cabinet), write what type of waste the bottle is for and write the start date (e.g. Acid Liquid Waste, Start date: 11/23/2015). Tape this paper to the bottle and place the bottle in the fume hood. Replace the sheet from the hood with a new one from the "Chemical Inventory Sheets" folder in the second drawer of the filing cabinet in the lab aide area.

b. The file path for printing new sheets when the stock is low is on the bottom of the sheet. Print new sheets and pre-cut them when the stock gets low. Cut new half sheets of blank paper (preferably paper from the shred boxes) and place them in the cabinet in the chemical storage room when the stock gets low.

26. If the benchkote lining the fume hood has become wrinkled or stained, replace it. Tape the paper in place with the rough, paper side up, and the smooth, shiny side down.
NOTE: Certain dropper bottles in the lab aide area are Teflon lined and are very expensive. These dropper bottles are to be used for reagents containing strong acids ONLY, and require the special, opaque caps. Currently, these bottles are being used for the Marquis, Mecke, and Froehde reagents. There are also amber dropper bottles in the lab aide area. These bottles are typically used for light sensitive reagents like the Sodium Nitroprusside reagent. All other reagents (with the exception of the Duquenois Levine reagents and the reagents stored in the refrigerator) are being stored in the smaller, clear dropper bottles. Best practice is to store the new lot# in the same type of container as the old lot#, unless a stability/safety issue has arisen, in which case the Chemistry section will have to re-evaluate its storage procedures. Refer to the photo below for the differences between each dropper bottle and its cap.

![Dropper Bottles](image)

Color Categories for Outlook Calendar

Red – Indicates expiration dates for reference materials/chemicals/reagents

Orange – Indicates that a reagent/solution needs to be made

Yellow – Indicates tasks that are performed quarterly, or less frequently, and other miscellaneous tasks (e.g. longevity studies, etc.)
Blue – Tasks that are performed once every two weeks, or monthly (temperature and balance checks)

Purple – Reagent QC checks
8.5 Title: LOGGING REAGENTS INTO LIMS

It is not necessary to complete each step in the order presented, but all steps must be completed.

1. Make the reagent/solution following the “Procedure for Making Reagents/Solutions” located in the Seized Drugs Training Manual, Chapter 04 General Chemistry Lab Aides and Processes, perform the QC check if applicable, and assign it an internal lot#. Internal lot #’s are assigned in Seized Drugs by using the letter “C” followed by the date in MMDDYY format, followed by a sequential number indicating the daily order in which reagents were made (e.g. the third reagent made on November 19, 2015 would have the internal lot # of C111915-3).

2. Open the Resource Manager Module in LIMS.

3. From the menu on the left, open LVMPD Forensic Laboratory → Resources → Reagents/Solutions → Chemistry.

4. Select the name of the reagent/solution you just made. If the name does not appear in the list, contact the LIMS Resource Manager Admin for the Chemistry Detail and request that it be added.

5. On the top left side of the screen, select “New Resource.”

6. In the window that opens, complete the following fields: Lot#, Date Created, Created By, Expiration Date, and Description. The description field should contain the name of the reagent/solution with the lot# in brackets (e.g. Marquis [C060514-2]). Refer to a resource that has already been added to see an example.

7. Click the “Components” tab, then choose “Add Components” from the ribbon. Choose the chemicals you used to make the reagent from the selected drop down menus. Only the compounds that are listed in the “Chemicals” folder will be available. You will not be able to add water from the ELGA system or reference materials as components. Also, if you are making a dilution of known concentration or a validation mix, do not add the solvent that you used as a component. You will list these items on the templates described in the following steps. This applies to dilutions of reference materials that are at a known concentration, and validation mixes ONLY. All other reagents/solutions must include the solvent(s) as components in LIMS as well as on the recipe templates in LIMS.

8. Save and Close the Resource Details Window.
9. For a reagent/solution that requires QC checks at regular intervals and the Marquis reagent, use the specialized template for that reagent/solution located in Qualtrax. For a reagent/solution that does not require QC checks at regular intervals (e.g. 50% NaOH), with the exception of the Marquis reagent, use the “General Reagent & Solution Prep Log” template located in Qualtrax.

10. Open the applicable template and complete all necessary fields including but not limited to the reagent/solution name, concentration (if applicable), technician, lot#, prep date, each component with manufacturer, lot#, and quantity used, total quantity prepared, expiration date, initial QC check date and results (if applicable).

11. Save this completed form as the name of the reagent followed by the internal lot# in [ ] and “Initial Preparation” (e.g. Mayer [070115-3] Initial Preparation). You can look at previous lot #’s in LIMS for an example.

12. Back in the LIMS system, select “Import” from the ribbon of the “Manage Files” window (window may be titled “Object Repository – Instance: ####”).

13. Find the file you just created and select “Open.” On the next screen, make any changes or notes in the Description field if necessary, then select “Save and Close.” Double click the file you just uploaded to open it and ensure that it is the correct form. If it is the correct form, close the document, highlight it from the list, and select “Approve.” If it is not the correct form, highlight it and select “Delete.” Begin again to upload the correct file. Close the Object Repository window, and Save and Close the Resource Instance Details.

14. Double check all the entries made for this resource for accuracy and consistency. Save and Close the “Resource Instance Details” window.
8.6 Title: QUALITY CONTROL CHECKING REAGENTS

1. **Always** follow Good Laboratory Practice (GLP) and all safety procedures outlined in the Forensic Laboratory Quality Manual, the Forensic Laboratory Safety Manual, the Seized Drugs Technical Manual, and the Seized Drugs Training Manual.

2. Be sure you have a clean, uncluttered working space. Use of the lab aide fume hood is ideal for reagent preparation; however, certain time sensitive tasks or circumstances involving casework may take precedence. If this is the case, you may wait (for a short period of time), or move to another fume hood. Ensure that you have an uncluttered workspace. Move existing items stored inside the fume hood to another fume hood if necessary. Be sure to replace the original items back into the appropriate fume hood when you are finished.

3. Collect all bottles of the current lot#. Collect the “Reference Materials for Reagent QC” box from CS refrigerator #6 desiccator in the Chemistry vault.

4. Get the most current recipe from the electronic version of the Seized Drugs Technical Manual in Qualtrax. Be familiar with all the content in the SDS of each ingredient in the reagent and proper disposal procedures. Keep in mind that printed versions of the Seized Drugs Technical Manual are not to be kept for future reference. A laptop is available near CS Hood#6 for viewing electronic documents.

5. If this is the first monthly QC check being performed on the reagent lot#, open the appropriate QC check form from Qualtrax (LVMPD → Forensic Lab Forms → Controlled Substances → Reagent QC). Each color test reagent has its own specialized template. At a minimum, enter in the lot number(s), the QC check date, the analyst’s p#/initials, the manufacturer and lot# of each reference material used, and the observed reaction.

6. Save the document to your desktop using the name of the reagent, followed by the lot# in brackets, then “QC Checks” (e.g. Cobalt Thiocyanate [C040417-1] QC Checks). For multi-part reagents (Chen’s, Dille-Koppanyi, etc.), the name of the file will show all applicable lot#’s (e.g. Chen Reagent [C081915-2] [C081915-3] [C081915-4] QC Checks). If this is not the first monthly QC check being performed on this lot#, check out the working document from Resource Manager in LIMS. NOTE: The Duquenois-Levine reagent uses a new form for every QC Check.
7. All color test reagents must be QC checked on schedule and immediately prior to expiration, disposal, or consumption. The final check prior to disposal ensures that the chemicals used to prepare the reagents were still viable, as some chemicals require a re-test. Each bottle must be QC checked at every interval, but can be logged on the same QC check form as long as all bottles give the same reaction.

8. A QC check must be performed with each of the reference materials listed under the Quality Control section of each recipe. The names of these reference materials are also pre-printed on the specialized templates.
   a. FreshPLATEs are available in CS Hood #6 and can be used to check multiple bottles at a time. Label the wells if necessary to prevent confusion.
   b. For the Mayer Reagent, a dark colored ceramic spot plate located in the drawer labeled “Spot Plates” in the lab aide area should be used for the QC check(s). FreshPLATE wells that have been colored with a black marker will also work, provided a blank check is also performed to ensure there are no interferences from the black marker.

9. Place a very small amount of each reference material to be tested into each appropriate well. The amount of reference material needed is no more than what can fit into the very end of a glass pipet (see photo below). Take great care not to cross contaminate the wells, and be aware that static forces may inadvertently cause some reference material to “jump” into another well. If you notice that this is occurring, DO NOT attempt to wipe the reference material out of the well. Use another well, or use a second spot plate. Some reagents are better performed in a test tube. This will be indicated in the recipe.

10. Follow the Testing Instructions in the recipe by placing drop(s) of the reagent(s) into each of the wells containing reference material. Take care not to contaminate the reagent dropper top or to splash into other wells. Ensure that all the reagent has fallen back into the bottle before replacing the dropper cap so as to prevent the collection of reagent around the outside of the cap and bottle. This can cause staining and discolorations that will be difficult to remove when it is time to clean the bottles. If you use a pipet or glass dropper to dispense the reagent, be sure not to touch the spot plate, as you may run the risk of contaminating the entire bottle.

11. Record your results on the appropriate electronic form and save the form.
   Compare your results to the results listed under the Quality Control section of the
recipe. Any deviations require immediate attention and may require that the lot be taken out of service.

a. If results were other than expected, repeat steps 7 through 9 for those tests. If contamination is suspected, repeat the steps using only one reference material per FreshPLATE.

b. If the results still deviate from what is expected, immediately notify the Chemistry Manager/Designee or a Forensic Scientist II. Further appropriate action will be determined. It is likely that a new batch of reagent will need to be made. If this is the case, follow the Procedure for Making Reagents/Solutions.

12. If a reagent level is low, check with all analysts to determine if a new batch is needed. Reagent needs are based on casework needs which are always changing. If a new batch is needed, follow the Procedure for Making Reagents/Solutions. Do not wait until the next QC check to determine if a new batch should be made. If the reagent level in one or more bottles is low, you may pour reagent from one bottle into another bottle, provided all bottles have first passed the QC check.

13. If all QC checks pass, dispose of all generated waste in the appropriate containers, per the Disposal section of the recipe and SDS. Indicate which chemical(s) you are disposing of in the liquid waste stream on the sheets on the fume hood sash. Be sure to check the SDS for incompatibilities before disposing. Not all acids play well together, and some should be containerized separately prior to placing in the acid bucket. Be aware that some chemicals should have their own bucket entirely.

14. Before returning each reagent bottle to its storage location, check each bottle for drips, and check labels for stains, legibility, clarity, and completeness. Clean, replace, and correct as needed. Check that the appropriate GHS labels are present, affixed to the bottles, and are not falling off. If necessary, use a piece of tape to secure them to the outside of each bottle. Return each reagent bottle to the original storage location from which you retrieved it.

15. Once the QC checks are complete, place any controlled reference material vials that were used to perform the QC checks into your appropriate re-weigh box and notify another analyst that items need to be reweighed.

16. If you notice that a reference material in the quality control check reference material box is low (there may not be enough to perform the next QC check), dispose of that reference material vial and create a new one using a new, empty, clean GC/MS vial.

a. If it is a controlled substance, have another FSII witness the disposal and properly annotate the disposal in the Chemical Inventory. Be sure to add a label with the correct manufacturer and lot# to the new vial. Place both the original reference material bottle that you used to create a new QC check vial, and the new QC check vial into your reweigh box to be checked/logged into the inventory.

b. If it is not a controlled substance, place the original reference material bottle back into its proper location as dictated by the electronic inventory,
and place the new QC check vial in your box to be logged into the inventory.

c. Notify another analyst that items need to be reweighed/logged and tell them that a QC check vial was remade. Do not wait until next month to remake a new vial, as there may not be enough for the next QC check. If the main stock of primary reference material is low, follow the procedures for ordering reference materials.

d. If the main stock of secondary reference materials is low, you may be required to follow the Department Drug Related Operations procedures outlined in the CS Technical Manual in order to replenish the stock. If necessary, notify the Chemistry Manager/Designee.

17. Once everything has been disposed of, empty the dry waste buckets from the fume hood into the blue barrels in the chemical storage room. Mark the list of chemicals on the blue barrel in the chemical storage room accordingly. Wipe off the start date on the bucket and replace it with today’s date. If the liquid waste container in the fume hood is full, cap it, write the end use date on the label, and place it in the appropriate blue barrel in the chemical storage room. Mark the chemical list on the blue barrel accordingly by comparing the list from the fume hood sash to the list on the blue barrel (remove the sheet from the hood sash and take it with you). After verifying that all the appropriate chemicals have been checked on the list on the blue barrel, dispose of the sheet from the hood sash in the trash.

18. Create a new liquid waste bottle by grabbing an empty 1 L bottle (or 4 L if 1 L bottles are not available) from the cabinet in the chemical storage room. On a blank piece of half paper (pre-cut paper should be in the same cabinet), write what type of waste the bottle is for and write the start date (e.g. Acid Liquid Waste, Start date: 11/23/2015). Tape this paper to the bottle and place the bottle in the fume hood. Replace the sheet from the hood with a new one from the “Chemical Inventory Sheets” folder in the second drawer of the filing cabinet in the lab aide area. The file path for printing new sheets when the stock is low is on the bottom of the sheet. Print new sheets and pre-cut them when the stock gets low. Cut new half sheets of blank paper (preferably paper from the shred boxes) and place them in the cabinet in the chemical storage room when the stock gets low.

19. If the benchkote lining the fume hood has become wrinkled or stained, replace it. Tape the paper in place with the rough, paper side up, and the smooth, shiny side down.

20. After completing the QC section on the appropriate form, the QC check must be entered into the Outlook Calendar and uploaded into the LIMS.
   a. In the Outlook Calendar, open the appropriate QC check appointment on today’s date by double clicking it, and add the word “Complete” at the beginning of the Subject line. If this reagent/solution requires QC checks at regular intervals, go to the date of the next required QC check and create an All Day Event. In the Subject line, enter in “QC XXX” with XXX indicating the name of the reagent/solution. Assign this appointment to
the purple category. QC checks should not occur on a weekend, holiday, or a Friday so choose the previous working day.

b. If this is the first monthly QC check performed on this lot#, open the Resource Manager Module in LIMS, and from the menu on the left, open LVMPD Forensic Laboratory → Resources → Reagents/Solutions → Chemistry. Select the name of the reagent/solution you just checked. Double click on the appropriate entry in the grid. In the Resource Instance Details window, choose “Manage Files” in the ribbon. In the Object Repository window that appears, select “Import.” Choose the appropriate file and select “Open.” On the next screen, make any changes to the title of the file, or notes in the Description field if necessary, then select “Save and Close.” Double click the file you just uploaded to open it and ensure that it is the correct form and that it contains your edits. If it is the correct form and it is complete, close the document, highlight it from the list, and select “Approve.” If it is not the correct form or all of the edits are not present, highlight it and select “Delete.” Make the necessary edits if necessary, and begin again to upload the correct file. Close the Object Repository window, and Save and Close the Resource Instance Details. For multi-part reagents (Chen’s, Dille-Koppanyi, etc.), the QC check will have to be uploaded to the Object Repository for each lot#. Delete the copy that was saved on your computer.

c. If this is not the first QC check performed on this lot #, check the QC log form back in to the same location from where it was checked out in the Resource Manager in LIMS. Open the Resource Manager Module in LIMS, and from the menu on the left, open LVMPD Forensic Laboratory → Resources → Reagents/Solutions → Chemistry. Select the name of the reagent/solution you just checked. Double click on the appropriate lot # in the grid. In the Resource Instance Details window, choose “Manage Files” in the ribbon. In the Object Repository window that appears, highlight the name of the file that was checked out. Choose “Check In” in the ribbon. Double click the file to open it and ensure that it is the correct form and that it contains your edits. Close the document, then save and close all windows for that lot#. For multi-part reagents, open the Resource Instance Details for the other lot #(s) one at a time and select “Manage Files”. Highlight the appropriate document and select “Update” from the ribbon. Select the appropriate document from your computer. Double click the file to open it and ensure that it is the correct form and that it contains your edits. If it is not the correct form, repeat the steps to “Update” the document with the correct form.

Repeat the procedure for any other QC checks that need to be completed this day.
8.7 Title: RECEIVING CHEMICALS

It is not necessary to complete each step in the order presented, but all steps need to be completed.

1. This procedure is for receiving chemicals only. Refer to the “Procedure for Verifying New Reference Materials” if a reference material was received.

2. Ensure that the laboratory received the correct item and quantity that was ordered. Write the received date, and your P#/initials on each received container.

3. Compare the packing slip to what was received. If everything checks out, write the received date and your P#/initials on the packing slip and give to the Sr. LEST. If items are missing, bring it to the attention of the Chemistry Manager/designee and Sr. LEST.

4. Obtain the Certificate of Analysis (C of A) and the MSDS/SDS from the manufacturer (via email, through their website, etc.). Save an electronic copy of each for importing into LIMS in step 7.f and 7.g.

5. Write the expiration/retest date on each received substance container. In the event that an expiration/retest date is not indicated (either on the container or C of A), check the folder located at H:\CB\Forensics\General\Chemical Inventory (MSDS)\Expiration Date Letters to see if a declaration from the manufacturer has been obtained in regards to the stability of the substance. If no expiration/retest date is indicated, “Exp: Until Consumed” or “Exp: U/C” shall be written on each received container or placed on each container using a sticker label.

6. Open the shared Chemistry calendar in Outlook. Find the expiration date of the chemical(s) you just received. Double click in the cell for that date to create an event. In the Subject line, enter, “Exp: [name of chemical][manufacturer and lot#]” (e.g. “Exp: CHCl3 [Avantor #K05B06]”). Check the “All day event” check box. Select “Categorize” from the ribbon and assign this to the red category. Save and Close. Both expiration and retest dates need to be entered into the calendar.

7. Make a new entry for the substance in the LIMS Resource Manager:
   a. Open Forensic Advantage
   b. Click on Resource Manager Module
   c. Click LVMPD Forensic Laboratory → Resources → Chemicals → Select the correct letter range depending on the substance → Select the correct sub-letter range depending on the substance.
NOTE: If the substance and lot number already exist in LIMS Resource Manager, the most recent MSDS/SDS and C of A are uploaded, and the storage recommendations have not changed, then no action is needed in this program, continue to step 8. If the storage recommendations have changed, adjust the LIMS entry by opening the details for that lot# and adjusting the “Location” field accordingly (remember to make the correction in the Excel inventory also). If you have a more recent version of the MSDS/SDS and/or C of A, upload it to the object repository for that lot# (see steps f and g below).

d. Select “New Resource”

e. In the window that opens, complete the following fields: Lot#, Received, Location, Asset ID (enter the manufacturer’s name), Expiration Date, Manufacturer and Description. The description field should contain the name of the substance with the mass/volume of the substance (e.g. Methanol – 4L). For consistency, the terminology for the location field should mimic the terminology used in previous entries, preceded by the word “CHEM – “ This will indicate that the substance is stored in the Chemistry Detail. Refer to a resource that has already been added to see an example. Retest dates should be added in the Expiration Date field. Click save and close.

f. Upload the C of A and MSDS/SDS (from step 3) into the LIMS Resource Manager for the received substance.
   i. Re-open the resource that is being added to LIMS
   ii. Click on the Manage Files button near the top of the window
   iii. Click Import
   iv. Select the C of A and MSDS/SDS from the location the files were saved in step 3
   v. Update the Name and Description fields if necessary, click Save and Close
   vi. Open the files to double check that the correct files were imported. Close the files.
   vii. With the imported files highlighted, click Approve. Close the window. Click Save and Close

g. Give a copy of the MSDS to the Lab Aide for filing.

8. Place the containers in the most appropriate storage location, per the manufacturer’s recommendation. This information can be found on the bottle, MSDS/SDS and/or the C of A.
It is not necessary to complete each step in the order presented, but all steps need to be completed.

1. Always follow Good Laboratory Practice (GLP) and all safety procedures outlined in the Forensic Laboratory Quality Manual, the Forensic Laboratory Safety Manual, the Seized Drugs/Trace Materials Technical Manuals, and the Seized Drugs/Trace Materials Training Manuals.

2. When a material has been consumed (consumed for this purpose means unusable quantities remain), contaminated, degraded, expired, etc. the product and/or container must be disposed of in the appropriate manner. For proper disposal of the material consult one or more of the following:
   a. the disposal log sheets that are located on the fume hood sashes and/or on the disposal barrels in the Chemical Storage/Waste room
   b. the Material Safety Data Sheet/Safety Data Sheet (MSDS/SDS) of the product
   c. the Chemistry Laboratory Manager or hazardous materials liaison
   d. the manufacturer of the product
   e. the hazardous materials transporter for guidance on disposal (currently H₂O Environmental)

3. Be sure to check all work areas and benches for any transfer containers, if applicable. If there are working solutions on analysts’ benches, ask or notify the analyst(s) that the lot # is being disposed of. If a lot # has expired, dispose of all containers of that lot # (e.g. color test reagents). If one container of the lot# is empty, but other containers are still being used, it is not necessary to dispose of all containers (e.g. solvent bottles). Contamination and degradation issues will be handled on a case by case basis. Discuss these issues with the Chemistry Detail/Manager.

4. Dispose of the consumed or expired material into the proper waste stream. Never pour or place any chemicals in the laboratory sinks. The only compounds that should go down the sink drains are soap and water.

   NOTE: Be sure to check all work areas for any transfer containers. Dispose of the original container as well as all transfer containers, if necessary.
   a. Solid/liquid reference materials and chemicals / acid/base solutions:
i. If disposing of a controlled substance, have an analyst/Manager assigned to the Chemistry Detail witness the disposal

ii. Rinse out the contents into the appropriate liquid waste stream receptacle, then place the entire container into the appropriate solid waste stream receptacle. Use methanol or a similar less-reactive solvent to perform the rinse.

iii. Update the log sheet(s) to reflect the addition of the material to the receptacle(s)

iv. For reference materials, check freezer CS #5 for any stock solution vials. Dispose of any stock solution vials that you find for the applicable lot number(s).

b. Liquid solvent materials:

i. For small volumes, pour the material into the appropriate liquid waste bottle in any of the fume hoods and update the waste log sheet to reflect the addition of the material to the receptacle.

   1. Leave the empty bottle uncapped in a fume hood until completely dry
   2. Replace the cap and put the bottle in the cabinet labeled “Empty Bottles” in the Chemical Storage room to be used later as hazardous material receptacle

ii. For large volumes, place the entire capped container (including contents) into the appropriate waste stream receptacle (e.g. Solvent Liquids blue barrel, or Acid Liquid blue barrel in Chemical Storage/Waste room). Update the log sheet on the barrel to reflect the addition of the material

   NOTE: “Small volumes” and “large volumes” are relative terms meant to be used at the discretion of the person disposing of the material in order to keep exposure, splashing and spilling to a minimum. Your safety and the safety of others is most important.

5. Archive the material and lot number in the LIMS Resource Manager so other analysts will not be able to add this lot number to their worksheets.

   a. Navigate to the appropriate compound and lot number
   b. Double click the appropriate lot# to open the Resource Details window
   c. Select the “Archived” check box
   d. In the Comments section, type the same information that you just entered into the Excel Inventory (e.g. Disposed 3/18/2014 M14485N; Witnessed by K8702N). Witnesses are only required when disposing of controlled reference materials.
e. Click Save and Close

6. If any waste stream receptacle is full, refer to the “Procedure for Replacing Hazardous Material Waste Receptacles.”

7. If the supply of the compound in the laboratory has been depleted or is low, be sure to order more. If you are unsure on the amount, grade, manufacturer, etc. to order, confer with a Forensic Scientist II or Chemistry Laboratory Manager to make certain the correct material and quantity is ordered.
8.9 Title: CHANGING COMPRESSED GAS CYLINDERS

HOW TO READ AND CONTROL THE PRESSURE:

The regulators are equipped with two gauges; one measures the cylinder pressure, the other measures the delivery pressure, also known as the line pressure. The gauge nearest the cylinder valve measures the cylinder pressure; the gauge farther away from the cylinder measures the delivery pressure.

It is common practice to measure pressures in units of psi (pounds per square inch); the readings for psi are located on the inner track of the gauge.

Once the cylinder valve has been opened, the cylinder pressure gauge will steadily drop over time as the cylinder slowly empties. It is important to monitor this pressure and replace the cylinder before it is completely empty.

The delivery pressure is set using the black knob located beneath the gauges. This pressure has already been set and should not need to be adjusted. Therefore, unless the cylinder is completely emptied, the delivery pressure reading should not change.
REPLACING A GAS CYLINDER

It is not necessary to complete each step in the order presented, but all steps need to be completed.

When changing compressed gas cylinders, follow all safety procedures outlined in the Forensic Laboratory Handbook and all safety practices stated in the Gas Regulators publication from California State University located in the Seized Drugs Training Manual.

1. Remember that replacing a cylinder temporarily interrupts the flow of gas to the instrument(s). Before proceeding, ensure that no instruments are running and no analyses will be affected by replacing the cylinder.

2. When a cylinder is ready to be replaced, retrieve a full cylinder, the open-ended wrench, and the Compressed Gas Tank Log book from the gas cylinder storage area by the back door.
   a. Verify the contents of the cylinder on the label.
   b. Transport the gas cylinder using the hand cart provided. Be sure to chain the tank into place prior to transporting.
   c. Back in the lab, position the hand cart in close proximity to where the new gas cylinder will be placed.

3. Close the cylinder valve of the empty gas cylinder. Follow the pigtail from the regulator to the copper line. Locate the plug valve (shown below). Close the plug valve by turning the green knob so that it is perpendicular to the copper line and the flow of gas. The image below shows the valve in the "open" position.

4. Using the appropriate, open-ended wrench, loosen the hex nut that secures the regulator to the cylinder and remove the regulator from the cylinder. Never use channel locks or pliers on the regulator or cylinder as this will damage the fittings. Avoid using a crescent wrench as this too can damage the fittings.

5. Place the regulator down on a secure surface. Take care not to cause undue tension to the gas lines to avoid weakening the copper lines and connections.

6. If there is a valve cap available for the empty gas cylinder, securely fasten it. If there is a ring on the tank indicating whether it is empty or full, ensure that the ring is set to show that the tank is “empty.” Unfasten the belt or chain holding the empty gas cylinder in place and move the empty gas cylinder to the side.

7. Remove the new gas cylinder from the hand cart and secure the belt or chain around it once in place.
8. Move the empty cylinder onto the hand cart and secure it using the chain attached to the cart.

9. If there is a valve cap on the new gas cylinder, remove it and place it on the table next to the cylinder (or use it to cover the valve of the empty cylinder).

10. If there is a cylinder valve plug, remove it.

11. Inspect the cylinder valve and threads for damage or contamination.
   a. If either is present, do not continue. Transport the cylinder to the storage area marked “Empty” and place a sign on the cylinder to indicate that there is a problem with the gas cylinder. Inform the Laboratory Aide/designee that we received an unusable gas cylinder. Retrieve a new gas cylinder and begin again.

12. Attach the regulator to the new tank by carefully hand-threading the hex nut onto the regulator to ensure a secure fit and to avoid stripping the threads of the nut. The connection should be easily threaded. If it is not easy, do not force it. Unscrew and try again.

13. Once the hex nut has been securely hand threaded, use the appropriate, open-ended wrench to further tighten the connection securely.

14. Once the hex nut has been sufficiently tightened, open the cylinder valve on the new gas cylinder.

15. Check all connections for leaks using an approved leak detection solution (e.g. Snoop®). Leaks are indicated by visible bubbling.
   a. If there is bubbling or an audible gas leak, tighten the appropriate connection(s) and recheck. The connection is secure once the bubbling has subsided.
   b. If the bubbling continues despite tightening, close the cylinder valve and remove the regulator. Obtain Teflon tape located in the Chemistry Detail.
      i. Before applying Teflon tape, inspect the threads on both the cylinder and the regulator and if necessary, clean the fitting to remove any dirt or thread sealant that remains on the threads.
      ii. Apply the Teflon tape starting at the second thread, as shown below.
iii. Ensure that the tape is wrapped in the direction of the thread spiral, pulling tightly on the end of the tape so that the tape conforms to the thread.
iv. Apply two overlapping layers of Teflon tape.
v. Cut off the excess tape and press the end firmly into the threads. Return to step 16.

16. Once the cylinder has been opened and no leaks are detected, open the plug valve (found immediately after the regulator) by turning the green knob so that it is parallel to the copper line and the flow of gas. Complete all fields in the Compressed Gas Tank Logbook, which include but are not limited to: type of gas, lot number, location of the cylinder, initial pressure reading (using psi units), initials and date.
   a. If the gas cylinder is connected to GC/MS instruments, ensure that the temperature and pressure for the front and back inlets for all applicable instruments are on.
   b. If the pressures shut down, turn them back on and ensure that the instrument holds pressure.

17. Return the empty gas cylinder to the storage area marked “Empty” and place the open-ended wrench, hand cart and the Compressed Gas Tank Logbook back to their proper storage locations.
Estimating the Uncertainty of Measurement

All members of the Seized Drugs Unit will be responsible for implementation of the uncertainty of measurement plan. The determined uncertainties are maintained in the Chemistry Laboratory Manager’s office. Volumes are not reported, therefore the estimated uncertainty of measurement was not determined for volumes.

Measurement of Uncertainty Budget

- Estimating the uncertainty of measurement will be determined by using the Measurement of Uncertainty Budget template located in Qualtrax under Forensic Lab Forms\Seized Drugs\Measurement Uncertainty Budget
- The uncertainty budget shall attempt to identify all the components of uncertainty and make a reasonable estimation.
- The uncertainty budget for a stated procedure will include Type A and Type B uncertainty components.
- All uncertainty components must be quantified in units that represent the measurement values.
- Components that are considered when determining the uncertainty of measurement for drug weights
  - Single vs. multiple items
  - Taring of a weighing vessel as a separate weighing operation
  - Aggregate weighings
  - Incomplete recovery of material from packaging
  - Balance selection (readability, capacity, calibration uncertainty, repeatability, linearity, buoyancy, temperature effects, sample loss in transfer)
  - Balance operation (sample placement on pan and corner loading)
  - Environmental conditions
- Components that are considered when determining the uncertainty of measurement for drug purity
  - Sample homogeneity
  - Sample preparation (sample size, matrix effects, solubility)
  - Analytical technique
  - Reference material (purity of standard)
  - Equipment and instrument properties (glassware, pipettes, balances, chromatographs)
  - Concentration of analyte
  - Environmental conditions
- Uncertainty components should not exceed two (2) significant figures.
- Calculations used to estimate the uncertainty of measurement and the combined uncertainty should be rounded up, to be conservative.
The rounded uncertainty must be reported to the same level of significance as the measurement result.

When combining uncertainty components, the units of each uncertainty value should be expressed in the same units.

The measurement of uncertainty budget will be re-evaluated when a major component is changed within the budget.

- Examples of major components
  - Change in the measured uncertainty determined by external calibration vendors
  - New instrument or equipment
  - New analytical technique
  - New Forensic Scientist

Type A Uncertainty Components

- Empirical data from within the laboratory using a measurement process evaluated by statistical methods (e.g. standard deviation).
- Repeatability
  - Repeated weighing of the same load under the same measurement conditions;
  - Calculate the standard deviation (σ) from a series of repetitive measurements of a stable measurand (this is best determined by historical data of a large population of repeated measurements).

Type B Uncertainty Components

- Uncertainty data derived from techniques other than repeated analyses and statistical calculations.
  - Examples: Uncertainties from calibration reports, Manufacturer’s specifications, Resolution, Readability

- These components must be made to ensure that the quantified uncertainties represent the equivalent of one (1) standard deviation. (e.g. manufacturer reports may report an expanded uncertainty, not equivalent to one (1) standard deviation)

Uncertainty Plan Associated with Drug Weights

- A specific measurement process will be defined.
- A measurement of uncertainty budget will be completed for each balance.
  - Each budget will include Type A and Type B uncertainty components that will be identified.
  - Type A uncertainty component will be the standard deviation (σ) of approximately 100 measurements of one (1) weight.
  - Type B uncertainty evaluation will be based on the information associated with:
    - reference standards calibrated by an external laboratory;
    - environmental parameters;
    - eccentric loading;
    - manufacturer’s published quantity values;
    - calibration certificate(s) and incorporation of drift;
    - limits deduced through personal experience.
Type B uncertainties without a confidence level will be treated as rectangular distributions. The standard deviation for this type of distribution is calculated using $\sigma = a/\sqrt{3}$.

- The combined uncertainty of uncorrelated components for each balance will be calculated using the root sum square (RSS) technique.
- Static or dynamic weighing techniques will be included in the expanded uncertainty calculation.
- Calculate the expanded uncertainty with a confidence level of approximately 95% by using a value of $k=2$.
- Report the results in the format of $y +/- U$ and the units of $y$ and $U$ to be consistent.
- The rounded uncertainty must be reported to the same level of significance as the measurement result.

### Reporting Uncertainty of Measurement for Multiple items
- Multiple items added together for a total weight
  - When multiple weights are added together to calculate a total weight, the uncertainties associated with each individual weight must be determined and included when reporting the total uncertainty. The root sum square (RSS) technique will be used for calculating uncorrelated uncertainties. When the same balance is used to determine the weights of multiple samples, then a correlated uncertainty will be determined. See the example for this calculation.

**EXAMPLE:**

- Six (6) plastic bags each containing plant material. The net weight of each plastic bag contents will be determined and summed. The same balance is used to determine the net weight and a static weighing technique is used. The combined uncertainty is 0.48 g, and is determined by summing the uncertainty from each measurand and multiplying by two (2) because of static weighing. The estimated expanded uncertainty of measurement for the balance used is $\pm 0.04$ g with $k=2$. ($U_{\text{combined}} \cdot 2 = U_{\text{expanded}}$)

\[
\begin{align*}
5.26 \text{ g} & \pm 0.04 \text{ g} \\
6.15 \text{ g} & \pm 0.04 \text{ g} \\
24.56 \text{ g} & \pm 0.04 \text{ g} \\
15.67 \text{ g} & \pm 0.04 \text{ g} \\
5.26 \text{ g} & \pm 0.04 \text{ g} \\
0.85 \text{ g} & \pm 0.04 \text{ g} \\
\end{align*}
\]

57.75 g + Calculated total expanded uncertainty

\[
{(0.04 \text{ g}) \cdot 6_{\text{number of measurements}} \cdot 2_{\text{static weighing}}} = 0.48 \text{ g}
\]

Total weight: 57.75 g + 0.48 g
Reporting Uncertainty of Measurement for multiple packages within an item weighed on multiple balances

- It is preferred that the same balance be used when weighing multiple packages within an item.
- There are times when different balances with different uncertainty of measurements may be used to weigh multiple packages within an item. If this occurs then the Root Sum Square (RSS) needs to be determined for the UOM, since the balances are uncorrelated. The weights will be added together with the total being truncated to reflect appropriate significant figures. The UOM determined by RSS will also be truncated to reflect the appropriate number of decimal places reflected by the summation of all the weights.

**SIGNIFICANT FIGURES**

Significant figures are defined as all digits that are certain plus one which contains some uncertainty.

Calculations:
The computation rules of significant figures are suggested to ensure that a calculated result contains only the number of digits justified by the experimental data. In addition and subtraction, the final result will only be as accurate as the least precise measurement and the rules will be applied at the end of that calculation, prior to proceeding with further mathematical operations. In multiplication and division, the final answer will contain the same number of significant figures as that of the value with the least numbers of significance. The rules of significant figures will be applied as the end of the entire calculation.

For calculations regarding entire items at a scene based on representative samples, the reported value will have the same number of significant figures as the laboratory measurement with the least numbers of significance. (Estimations for volumes at the scene and conversion factors are not considered to contain any significant figures because there is no measurement associated with that value.)

For example, a liquid sample taken from a scene was determined to be 35.269 g with a volume of 34.8 mL. If the estimated quantity at the scene was 1 gallon, the calculation would be as follows:

\[(35.269 \text{ g}/34.8 \text{ mL}) \times (1 \text{ gallon}) \times (3.785 \text{ L} / 1 \text{ gallon}) \times (1000 \text{ mL} / 1 \text{ L}) = 3,836.0104 \text{ g}\]

Following the rules of significant figures, the volume measured in the laboratory has the least number of significant figures - three. Therefore, the estimated weight of the sample at the scene could only be reported as 3,830 g.

**DEFINITIONS**

**Accuracy** – Closeness of the agreement between the determined result and the true value
Combined standard uncertainty - standard uncertainty of the result of a measurement when that result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with changes in these quantities.

Cornerload – Inaccuracy of the balance factor for objects that are not placed centrally on the balance.

Coverage factor - numerical factor used as a multiplier of the combined standard uncertainty in order to obtain an expanded uncertainty.

Dynamic weighing – Example: tare a weigh boat, add powder to weigh boat while still on the balance, read weight before removing the weigh boat and powder from the balance.

Eccentric loading error - Also referred to as "corner load error" or "off-center loading error". This is the change in the readout when the same object is placed in various positions on the weighing pan.

Expanded measurement uncertainty - expanded uncertainty product of a combined standard measurement uncertainty and a factor (coverage factor) larger than the number one.

Linearity – The capability of the balance to follow the linear relation between the load and the displayed value.

Measurand - quantity intended to be measured.

Measurement - process of experimentally obtaining one or more quantity values that can reasonably be attributed to a quantity.

Measurement procedure - detailed description of a measurement according to one or more measurement principles and to a given measurement method, based on a measurement model and including any calculation to obtain a measurement result.

NOTES
1 — A measurement procedure is usually documented in sufficient detail to enable an operator to perform a measurement.
2 — A measurement procedure can include a target measurement uncertainty.
3 — A measurement procedure is sometimes called a standard operating procedure, abbreviated SOP.

Measurement bias - systematic measurement error or its estimate, with respect to a reference quantity value.

Precision – Extent of mutual agreement between several measured values, independent of the true value.

Readability – The smallest difference between two measured values that can be read on the display.
**Rectangular, or Uniform Distribution** - In a rectangular distribution all values within a range between $a$ and $b$ are equally likely.

**Repeatability** – The ability of a balance to provide the same result for repeated weighings of the same load under the same measurement conditions

**Root sum square (RSS) technique** – Mechanism of calculating uncorrelated uncertainties. Each of the standard uncertainty values are squared and then added together prior to taking the square root of the sum.

**Standard uncertainty** - uncertainty of the result of a measurement expressed as a standard deviation

**Static weighing** – Example: tare a weigh boat, remove weigh boat from balance, add powder to weigh boat, place weigh boat and powder back onto balance, read the weight from the balance

**Systematic measurement error** - component of measurement error that in replicate measurements remains constant or varies in a predictable manner

NOTES
1 — The reference quantity value for a systematic measurement error is a true quantity value, or a measured quantity value of a measurement standard of negligible measurement uncertainty, or a conventional quantity value.
2 — Systematic measurement error, and its causes, can be known or unknown. A correction can be applied to compensate for a known systematic measurement error.
3 — Systematic measurement error equals the difference of measurement error and random measurement error.

**Type A** - evaluation of a component of measurement uncertainty by a statistical analysis of quantity values obtained under defined measurement conditions

**Type B** - evaluation of a component of measurement uncertainty determined by means other than a Type A evaluation of measurement uncertainty

**Uncertainty budget** - statement of a measurement uncertainty, of the components of that measurement uncertainty, and of their calculation and combination

NOTE
The uncertainty budget should include the measurement model, estimates and measurement uncertainties of the quantities in the measurement model, covariances, type of applied probability density functions, degrees of freedom, type of evaluation of measurement uncertainty, and any coverage factor.

**Uncertainty of measurement** - parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used

NOTES
1 — Measurement uncertainty includes components arising from systematic effects, such as components associated with corrections and the assigned quantity values of measurement standards, as well as the definitional uncertainty. Sometimes known systematic effects are not corrected for but are instead treated as uncertainty components.
2 — The parameter may be, for example, a standard deviation called standard measurement uncertainty (or a specified multiple of it), or the half-width of an interval, having a stated coverage probability.
3 — Measurement uncertainty comprises, in general, many components. Some of these may be evaluated by Type A evaluation of measurement uncertainty from the statistical distribution of the quantity values from series of measurements and can be characterized by experimental standard deviations. The other components, which may be evaluated by Type B evaluation of...
measurement uncertainty, can also be characterized by standard deviations, evaluated from probability density functions based on experience or other information.

REFERENCES:
International Vocabulary of Metrology – Basic and General Concepts and Associated Terms (VIM), 3rd edition. Final draft 2006-08-01.


Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) guidelines listed Supplemental Document SD-3 revision 2.

Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG) guidelines listed Supplemental Document SD-4 revision 0.
# LVMPD FORENSIC LABORATORY
## TECHNICAL PROCEDURES
### SEIZED DRUGS

## 10.1 Title: ABBREVIATIONS KEY

*Capitalization may vary*

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>ACN</td>
<td>(acetonitrile)</td>
</tr>
<tr>
<td>ATR</td>
<td>(Attenuated Total Reflectance)</td>
</tr>
<tr>
<td>BSTFA</td>
<td>(N,O-Bis(trimethylsilyl)trifluoroacetamide)</td>
</tr>
<tr>
<td>BZP</td>
<td>(Benzylpiperazine)</td>
</tr>
<tr>
<td>CCDA</td>
<td>(Clark County District Attorney)</td>
</tr>
<tr>
<td>CS or C/S</td>
<td>(controlled substance(s))</td>
</tr>
<tr>
<td>COA</td>
<td>(Certificate of Analysis)</td>
</tr>
<tr>
<td>conc</td>
<td>(concentrated)</td>
</tr>
<tr>
<td>CTC</td>
<td>Column Test Check</td>
</tr>
<tr>
<td>CTS</td>
<td>(Collaborative Testing Services)</td>
</tr>
<tr>
<td>DI</td>
<td>(de-ionized)</td>
</tr>
<tr>
<td>DIB</td>
<td>(Drug Identification Bible)</td>
</tr>
<tr>
<td>DMF</td>
<td>(dimethylformamide)</td>
</tr>
<tr>
<td>DMSO</td>
<td>(dimethylsulfone)</td>
</tr>
<tr>
<td>DXM</td>
<td>(Dextromethorphan)</td>
</tr>
<tr>
<td>Et₂O</td>
<td>(diethyl ether)</td>
</tr>
<tr>
<td>EV #</td>
<td>(Event Number)</td>
</tr>
<tr>
<td>ext</td>
<td>(extraction)</td>
</tr>
<tr>
<td>FS</td>
<td>(Forensic Scientist)</td>
</tr>
<tr>
<td>FID</td>
<td>(Flame ionization detector)</td>
</tr>
<tr>
<td>FTIR/ATR</td>
<td>(Fourier Transform Infrared Spectroscopy and/or Attenuated Total Reflectance)</td>
</tr>
<tr>
<td>Foxy</td>
<td>(Foxy Methoxy, 5-methoxy-N,N-diisopropyltryptamine (5-MeO-DIPT))</td>
</tr>
<tr>
<td>GC</td>
<td>(gas chromatography/gas chromatograph)</td>
</tr>
<tr>
<td>H⁺ or H</td>
<td>(acid)</td>
</tr>
<tr>
<td>IDAD</td>
<td>(Ident-A-Drug)</td>
</tr>
<tr>
<td>IPA</td>
<td>(isopropyl alcohol)</td>
</tr>
<tr>
<td>LIMS</td>
<td>(Laboratory Information Management System)</td>
</tr>
<tr>
<td>MeCl₂</td>
<td>(methylene chloride)</td>
</tr>
<tr>
<td>MDMA</td>
<td>(methylendioxymethamphetamine)</td>
</tr>
<tr>
<td>MDEA</td>
<td>(methylenedioxyethylamphetamine)</td>
</tr>
<tr>
<td>MDA</td>
<td>(methylenedioxymethylamphetamine)</td>
</tr>
<tr>
<td>MS</td>
<td>(mass spectrometry/mass spectrometer)</td>
</tr>
<tr>
<td>MSDS</td>
<td>(Material Safety Data Sheet)</td>
</tr>
<tr>
<td>MSTFA</td>
<td>(N-methyl-N-trimethylsilyltrifluoroacetamide)</td>
</tr>
<tr>
<td>MW</td>
<td>(Molecular Weight)</td>
</tr>
<tr>
<td>NCDA</td>
<td>(Nye County District Attorney)</td>
</tr>
<tr>
<td>NCS</td>
<td>(not a controlled substance)</td>
</tr>
<tr>
<td>NCS, Rx</td>
<td>(not a controlled substance, prescription required)</td>
</tr>
<tr>
<td>NCSD</td>
<td>(no controlled substance(s) and/or dangerous drug(s) were conclusively identified)</td>
</tr>
<tr>
<td>OH or OH⁻</td>
<td>(base)</td>
</tr>
<tr>
<td>OOS</td>
<td>(Out of Service)</td>
</tr>
<tr>
<td>OR</td>
<td>(Object Repository)</td>
</tr>
<tr>
<td>P2P</td>
<td>(Phenyl-2-propanone)</td>
</tr>
<tr>
<td>ppt</td>
<td>(precipitate)</td>
</tr>
<tr>
<td>PC</td>
<td>(Property Connect)</td>
</tr>
<tr>
<td>PCF</td>
<td>(propyl chloroformate)</td>
</tr>
<tr>
<td>POI</td>
<td>(Persons of Interest tab)</td>
</tr>
<tr>
<td>PTHIT</td>
<td>(Phenyltetrahydromidazothiazole)</td>
</tr>
<tr>
<td>QC √</td>
<td>(Quality Control check)</td>
</tr>
<tr>
<td>RFLE</td>
<td>(Request for Forensic Laboratory Examination)</td>
</tr>
<tr>
<td>RM</td>
<td>(Resource Manager or reference material)</td>
</tr>
<tr>
<td>RT</td>
<td>(retention time)</td>
</tr>
<tr>
<td>rxn</td>
<td>(reaction)</td>
</tr>
<tr>
<td>S or Supp</td>
<td>(supplemental)</td>
</tr>
<tr>
<td>sat.</td>
<td>(saturated)</td>
</tr>
<tr>
<td>SD</td>
<td>(seized drugs)</td>
</tr>
<tr>
<td>SDS</td>
<td>(Safety Data Sheet)</td>
</tr>
<tr>
<td>TFMPP</td>
<td>(Trifluoromethylphenylpiperazine)</td>
</tr>
<tr>
<td>TIC</td>
<td>(Total Ion Chromatogram)</td>
</tr>
<tr>
<td>TLC</td>
<td>(thin layer chromatography)</td>
</tr>
<tr>
<td>TMCS</td>
<td>(trimethylchlorosilane)</td>
</tr>
<tr>
<td>TPC</td>
<td>(N-trifluoracetyl-L-prolyl chloride)</td>
</tr>
<tr>
<td>UC or U/C</td>
<td>(Until consumed)</td>
</tr>
<tr>
<td>UR</td>
<td>(Unit Record)</td>
</tr>
</tbody>
</table>
Chemistry Computer Software Versions:

FTIR:
Nexus 670: OMNIC 7.1, Val Q (validation) no version
Nicolet 380: OMNIC 9.8.372, ValPro Qualification 2.5

GCMS:
CS #3
MSD Chemstation E.02.02
CS #13; 15; 16; 17 and Trace #6
MSD Chemstation E.02.01 or F.01.03
MassHunter GC-MS Acquisition B.07.06
MassHunter Workstation Software – Qualitative Analysis Navigator B.08.00

Microscope camera for Trace:
Leica Application Suite Version 3.8.0

Raman Microscope:
OPUS Version 7.2

XRF:
Thermo Scientific Niton Data Transfer Version 8.4.2

Portable Raman Devices:
TruNarc: TruNarc Admin Installer v1.5
TacticID: TID Software Drive Version 4.04
The manufacturer’s manual(s) for the following instruments are located within the Chemistry Laboratory.

- **GC/MS**
  
  *Hewlett Packard Chemstation, Understanding Your Chemstation ©1998.*
  
  
  

- **FTIR-ATR**
  

- **Raman Microscope**
  
  *Bruker Senterra User Manual. 2nd Edition © 2009*
  
  *Olympus BX51 System Microscope Instructions © 2011*
  
  *OPUS Spectroscopy Software Version 6 Manual II © 2006*

- **Portable Raman Device**
  
  *Thermo Scientific TruNarc User Guide © 2013*

- **Microscopes**
  
  
  *Leica E-Series User Manuals, Version February 2014*

- **Balances**
  
  *Operating instructions METTLER TOLEDO AG balances ©2000.*
  
  *Operating instructions METTLER TOLEDO PG-S balances (0.001g, 0.01g) ©1998.*
Operating instructions METTLER TOLEDO Excellence XS Precision balances ©2006.


- **Pipettes**


- **XRF**
  Thermo Scientific Niton Data Transfer Software Version 8.4.2 Niton XLt/p/i/XL2-XL3-FXL-DXL Resource Guide USB Driver
LVMPD FORENSIC LABORATORY
TECHNICAL PROCEDURES
SEIZED DRUGS

10.3 Title: REFERENCES


   
   1. Carboxylic acids, Section 18.1 through 18.4, pp.579-584
   2. Amines part I, Section 22.1 through 22.5, pp.727-731


   1. Carboxylic acids, Section 18.1 through 18.4, pp.579-584
   2. Amines part I, Section 22.1 through 22.5, pp.727-731


37. Merck Index.

   This is probably the single, most comprehensive publication available on the subject of pharmaceuticals. Individual monographs usually give information as to chemical structure, trade names, manufacturing procedures, physical and chemical properties, medical uses and toxicological data.

38. Physician's Desk Reference

   The PDR is a valuable piece of information because of its Product Identification Guide. Often a look at the PDR will reveal the tablet you received as an unknown. Unfortunately, only those products that the manufacturer wishes to advertise are listed and these constitute a small portion of the market.

“Clarke” is an exhaustive treatise on pharmaceutical and toxicological analysis, including TLC, GC, UV, IR, solubility data and extraction procedures.

40. Microgram.

The Microgram is a publication of the Drug Enforcement Administration. Its major function is to exchange information between DEA labs and state and local laboratories. New items or trends in drug abuse are reported, as well as technical papers on the identification and assay of drugs.


42. Modern Methods of Pharmaceutical Analysis - Schirmer, Volumes 1-3.

43. Mills, et.al., Instrumental Data for Drug Analysis, Volumes I - VI.

44. Schultes and Hofmann, The Botany and Chemistry of Hallucinogens.

45. Bailey and Rothblatt, Handling Narcotic and Drug Cases.

46. Feigl, Spot Tests in Organic Analysis.

47. DEA Logo Index.

48. Analysis of Drugs - DEA Publication.


50. I. Sunshine, Handbook of Analytical Toxicology, CRC.

51. I. Sunshine, Handbook of Mass Spectra of Drugs, CRC.

52. Willard, Merritt & Dean, Instrumental Methods of Analysis

53. W. H. McFadden, Techniques of Combined Gas Chromatography/Mass Spectrometry; Applications in Organic Analysis.


57. Controlled Substances Handbook, S. Cook, Inc.

58. Drozd, J., Chemical Derivitization in Gas Chromatography.
59. CND Analytical, Inc., Analytical Profiles, (P.O. Box 1527, Auburn, AL, 36831-1527) References on: Amphetamines and Related Phenethylamines Substituted 3,4 Methylendioxyamphetamine Cocaine, Local Anesthetics, and Common Diluents Precursors and Chemicals Methylaminorex and Analogs Narcotics Anabolic Steroids Hallucinogens Barbiturates


64. SWGDRUG Recommendations.