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3.1 Quality Assurance

The quality management system includes evidence handling, management practices, equipment, testing, and reporting. It also covers all procedures used in the analysis of seized drugs.

This chapter focuses on particular elements of the quality management system as they relate to the Controlled Substances Units.

Refer to other FSD Manuals for additional topics, including:

- Qualification of Personnel
- Proficiency Testing
- Safety and Security
- Facilities
- Evidence Control
- Internal audits
- Risk Assessment and Corrective Action

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3.2 Case Records

Case records will contain the results, report and sufficient information to facilitate identification of factors affecting the measurement result and to enable the laboratory activities to be repeated under conditions as close as possible to the original.

Original observations, data and calculations shall be recorded at the time they are made and identifiable with the specific task.

Technical records to support a laboratory report shall be such that another reviewer possessing the relevant knowledge, skills, and abilities could evaluate what was done and interpret the data.

The following will be included in all case records, as applicable:

- a) The date and identity of personnel responsible for each laboratory activity.
- b) The date and identity of personnel responsible for checking data and results.
- c) The equipment used.
- d) The instrument models and the operating parameters of the instruments used in analysis.
- e) All data produced.
- f) Both original and amended data, results, or reports.
- g) The date and identity of personnel responsible for amendments.

Abbreviations and symbols specific to FSD are found in [Appendix 3](#). The abbreviations and symbols listed may be used in case records to facilitate efficient note taking.

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3.3 Equipment Records

3.3.1 Introduction

This policy describes the types of records that are maintained for equipment in the Controlled Substances Unit. Equipment includes, but is not limited to:

- measuring devices and measurement standards
- reference materials
- reagents
- analytical instruments

This policy does not pertain to FSD-administered software applications including the LIMS.

3.3.2 Records

Records will be maintained for each piece of equipment which can influence laboratory activities.

Common equipment record-keeping formats:

- a) Logbook
- b) Electronic log
- c) Qualtrax workflow
- d) FSD Apps

Specific equipment record formats for analytical instruments or balances should be used when available. Examples: FS-45, FS-50, FS-51

3.3.3 Documentation

Equipment records are retained and include the following, where applicable:

- a) Identity (name) of equipment
- b) Manufacturer's name
- c) Serial number or other unique identification
- d) Software and firmware versions
- e) Current location
- f) Dates, results, and copies of reports and certifications of all calibrations, adjustments, validation, verification, acceptance criteria, and due date of next calibration.
- g) Maintenance plan and maintenance carried out to date
- h) Damage, malfunction, modification, or repair of the equipment
- i) Other details as required by any sections of the procedure manual specific to the equipment

Records for FSD-maintained instrumentation are on FSD Apps and in the instrument logbooks. Records for measuring equipment and standards are maintained in Unit logbooks and in Measurement Uncertainty workbooks. Information for reagents and reference materials is found in Unit logbooks or on Qualtrax.

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3.3.4 Verification and Calibration Records

Equipment shall be verified before being placed or returned into service.

- a) Verification and validation records of new equipment will be retained in Qualtrax.
- b) On-going quality checks or return to service checks may be maintained in the equipment logbook.

Verification records typically include the results of the verification tests and the data or reports generated along with a determination if the equipment is working properly.

Calibration records shall be maintained for each piece of equipment requiring calibration. Refer to [Appendix 3.3](#) and [CS-PM 4](#) for calibration requirements.

3.3.5 Out of Service Equipment

If equipment cannot meet the acceptance criteria for the applicable verification checks, it will be clearly marked as out of service. A corresponding entry will be made in the equipment record or logbook.

The record and/or logbook will be updated when maintenance is performed and when the equipment has been successfully verified. Unsuccessful attempts at verification following maintenance should also be documented.

3.3.6 Moving Equipment to a New Location

Equipment that is not meant to be regularly moved shall be transported in a manner that is safe for personnel and minimizes the potential for damage or contamination of the equipment.

Reference standards and reference materials must be transported in a manner that does not alter the relevant properties of the reference. Standard weights must be transported in the padded case used for storage.

Upon installation at the new location, equipment must be verified by a routine function check prior to use in casework. Balances must be calibrated prior to use.

Records of calibration and verification checks after moving equipment to a new location will be retained. When moved to a new laboratory, a copy of the original logbook and equipment records may be forwarded to the receiving laboratory for continuation upon receipt and verification. Original records may be retained by the originating laboratory.

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3.4 Drug Reference Materials

3.4.1 Introduction

Reference materials, in general, are materials with known or established qualitative or quantitative properties. Reference materials, reference collections, and reference data are important to demonstrating the validity of qualitative and quantitative test results.

3.4.2 Scope

This policy applies to drug reference materials used in the Controlled Substances Unit for the purposes of qualitative identification or quantitation. This policy also pertains to materials retained for training purposes.

3.4.3 Terminology

Reference Material is material, sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of qualitative or quantitative properties.

Certified Reference Material is a reference material that has a certified value that was established through metrologically valid procedures and is provided with an associated uncertainty.

External Reference Data is chemical or physical data obtained from a drug Reference Material that was generated external to the Michigan State Police Forensic Science Division.

Comparable Reference Data is reference data analyzed under the same or suitably similar analytical conditions as the test or case sample.

Drug Standard is a commonly used term that is synonymous with “drug reference material.” Drug standards are not the same as metrological “reference standards.”

Reference Standard is a measurement standard that is used routinely to calibrate or verify measuring devices. Also known as a working measurement standard.

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3.4.4 Ordering, Receipt, and Storage of Reference Materials

3.4.4.1 Ordering

Reference materials should be obtained from a provider accredited under ISO Guide 34 or to ISO/IEC 17034.

NOTE: The Vendors report in Qualtrax (ID 3453) may be filtered for accredited reference material providers by applying the Accreditation Type filter.

Containers of no more than 250 milligrams of any drug substance should be purchased.

3.4.4.2 Receipt of Reference Materials

Upon receipt of any drug reference material, the Unit Supervisor or designee shall initial and mark the container with date of receipt and assign a unique identifying number to each lot of material received.

The unique identifying number shall allow for different lot numbers and different bottles within the same lot to be distinguishable.

- a) Identical chemicals of different lot numbers or bottles should be given a sequential suffix to distinguish between lots and bottles. For example, the first bottle of cocaine reference material may be 10-01, the second 10-02.
- b) If a reference material is transferred to a second laboratory, the identifying number at the receiving laboratory shall include the common two-letter laboratory designation as a prefix to the identifying number. For example, Bridgeport Laboratory cocaine reference material 10-01 is identified as BP-10-01 if transferred to other laboratories.
- c) If the unique identifying number already includes the laboratory designation, no prefix is needed. For example, BP10-01 may be sufficiently unique to identify the material at all laboratories.

Each container or bottle of material shall also be marked with the unique identifying number when practicable.

3.4.4.3 Storage of Reference Materials

Reference materials shall be stored in a substantially constructed vault or safe, and according to manufacturer's recommendations.

Access to drug reference materials shall be granted to Controlled Substances Unit personnel only.

3.4.5 Reference Material Performance Requirements

All reference materials obtained by the laboratory shall be assessed to verify that the material is fit for purpose.

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The fit-for-purpose assessment shall be done on each lot of reference material.

The assessment of a reference material shall be done prior to, or alongside casework analysis, as appropriate.

3.4.6 Assessment for Qualitative Analysis

The acceptance of a reference material for qualitative work requires an assessment of chemical identity by in-house analysis.

The verification of chemical identity by analysis shall be accomplished by analysis and comparison of results to one or more of the following:

- a) Peer-reviewed published data
- b) Data produced by a laboratory accredited under ISO/IEC 17025
- c) Data produced from a previously verified reference material

If sufficient reference data does not exist, reference materials obtained from a provider accredited under ISO Guide 34 or ISO/IEC 17034 can be accepted as correct if the material is stored and used in accordance with the manufacturer's instructions.

The certificate of analysis provided by the manufacturer and manufacturer's accreditation certificate shall be retained in the reference materials logbook for materials verified solely based on manufacturer accreditation.

3.4.6.1 External References

Comparison to external reference data may be used where a reference material is unavailable.

External reference data shall be shown to be fit for purpose. The veracity of data shall be considered and assessed upon relevant factors including:

- a) Comparability of analytical conditions
- b) Origin of the data
- c) Validation or peer review of the data

The use of external reference data rather than a reference material shall be documented, and where applicable, limitations shall be expressed in the laboratory report.

Refer to Appendix 3.4-A for a listing of sources for external reference data.

3.4.7 Assessment for Quantitative Analysis

The acceptance of a reference material for quantitative analysis requires an assessment of purity or concentration, its associated uncertainty of measurement, and an assessment of chemical identity.

For quantitative determinations, different sources of reference material should be used for calibration and for quality control. Where this is not feasible, two different lots of the same source may be used. If this is not feasible, as a last resort, a single source of reference material will be sub-divided, and each part assigned a specific purpose.

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Quantitative reference materials obtained from a provider accredited under ISO Guide 34 or ISO/IEC 17034 can be accepted as correct if the material is stored and used in accordance with the manufacturer's instructions.

The certificate of analysis provided by the manufacturer and manufacturer's accreditation certificate shall be retained in the reference materials logbook for materials verified based on manufacturer accreditation.

For quantitative reference materials obtained from a provider not accredited under ISO Guide 34 or ISO/IEC 17034 the purity and/or concentration shall be verified by analysis.

3.4.8 Other Performance Requirements

All qualitative and quantitative analytical verification data shall be labeled with:

- a) The unique standard number.
- b) The source of the reference material.
- c) The lot number of the reference material.
- d) The date and time the data was collected.
- e) The initials of the scientist performing the assessment of the reference material.

3.4.9 Reassessment of Reference Materials

3.4.9.1 Qualitative Reference Materials

If the expiration date passes before a qualitative reference material is fully used, the material shall be reassessed, and the expiration date extended.

A reassessment for qualitative analysis shall include an assessment of chemical identity by analysis.

After verification of chemical identity, the expiration date shall be extended by not more than 1 year. Refer to the analytical guides related to specific chemicals for exceptions to the expiration date extension.

If the reference material is not fully used after the passage of the expiration date extension, the reference material shall be reassessed for qualitative analysis on an annual basis according to the Reassessment of Reference Materials procedure.

3.4.9.2 Quantitative Reference Materials

If the expiration date passes before a quantitative reference material is fully used, the material will be replaced.

3.4.10 Reference Material Assessment Test Methods

For qualitative assessment by analysis, at least one test with sufficient discriminating power shall be employed. Generally, a Category A technique (mass spectrometry or infrared spectroscopy) is sufficient.

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The chemical identity of the reference material being assessed will determine which technique(s) are suitable.

3.4.11 Inspection

Verification data shall be assessed using the acceptance criteria of the technique(s) used to generate the data. Refer to the relevant instrument procedure(s).

3.4.12 Rejection

If acceptance criteria are not met for either a qualitative or quantitative assessment of a reference material, the assessment of the reference material shall be repeated using the same method and instrumental technique.

If the repeated assessment by the same method and technique does not meet acceptance criteria for qualitative verification, a different technique shall be employed.

If the repeated assessment still does not meet the acceptance criteria for the test method:

- a) The Unit Supervisor shall be notified.
- b) The Unit Supervisor or designee shall notify the Technical Leader.
- c) The reference material shall not be used as a qualitative or quantitative reference.
- d) The reference material producer should be notified.

3.4.13 Quality Assurance

Reference materials shall be stored according to manufacturer's recommendations.

The use of a reference material in casework shall be documented in the case record by recording the unique identification number of the reference material. This documentation shall be correlated to the specific task in which the reference material was used.

If no expiration date is provided by the reference material provider of a qualitative reference material, an administrative expiration date of two (2) years from the date of original instrumental verification shall be assigned to qualitative reference materials. The reference material shall be reassessed prior to use after this date by following the procedure for Reassessment of Reference Materials.

If no expiration date is provided by the reference material provider of a quantitative reference material, an administrative expiration date of one (1) year from the date of original verification shall be assigned to quantitative reference materials.

3.4.14 Reference Material Records

A Reference Material Logbook shall be maintained by each laboratory for all reference materials held at that laboratory.

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An index sheet listing the reference materials held by the laboratory should be maintained in the front of the Reference Material Logbook.

Form FS-44 shall be used to document each reference material and shall be placed into the Reference Material Logbook when the material is assessed as suitable for use.

Each page shall be labeled with the unique identifying number of the reference material.

All pages for each reference material shall be sequentially numbered.

All fields should be completed to the extent possible.

The expiration date shall be recorded. An administrative expiration date shall be assigned when none is provided by the manufacturer. Refer to the Quality Assurance section of this policy. The extended expiration date(s) shall be recorded when applicable.

3.4.15 Use of Reference Materials

Before and after each use of any reference material, the gross weight of the container and its contents to two (2) decimal places shall be recorded in the Reference Material logbook on the page for that reference material. The date of the weighing and the analyst's initials are also to be recorded.

Any difference between sequential weighings of a reference material that exceed 0.1 g shall be accounted for and documented. The difference should also be reported to the Unit Supervisor.

When the reference material has been fully used, an entry noting that the material was depleted shall be placed in the comments section of the logbook page for that material.

The logbook pages for depleted materials shall remain in the reference materials logbook for a minimum of 5 years, or one full accreditation cycle, whichever is longer.

All empty containers of depleted reference materials shall be given to the Unit Supervisor or designee for disposal.

3.4.16 Training Materials

A Training Material Logbook shall also be maintained by each laboratory for all drug materials retained for training purposes. This logbook shall be marked "Training" and shall use a different unique identifying numbering scheme than that used for identifying analytical reference materials.

The FS-44 form shall be used to document training materials in the same manner it is used to document analytical reference materials.

Training materials shall not be used as analytical reference materials in casework.

3.4.17 Samples from Casework

The retention of samples from casework for intelligence or training purposes shall follow the procedure entitled "Retention of Samples from Casework" in Appendix 3.4.-B

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3.4.18 Reference Material Audit

At least once per year, a minimum of ten percent (10%) of the materials in the drug reference material collection shall be weighed for quantity verification by the Unit Supervisor. The weight, date, and the initials of the individual performing the audit shall be documented on the appropriate log sheets.

NOTE: It is recommended to focus this effort on materials with a large mass relative to that of the container to minimize the extent of primarily measuring the weights of containers.

A list of substances weighed as part of the reference material audit shall be maintained in the reference material logbook. An alternate repository, such as an audit document binder, is acceptable if the name and/or location of the alternate repository is stated in the reference material logbook.

3.4.19 Federal DEA Controlled Substance License Inventory

To comply with federal licensing, the DEA license registrant shall take a new inventory of all stocks of controlled substances on hand at least every two years. The biennial inventory may be taken on any date which is within two years of the previous biennial inventory date.

Federal regulations are written so as to exclude almost all the small quantities of reference materials encountered in the Controlled Substances Units. It is expected that the laboratory will not maintain any stock of controlled substances which will require a biennial inventory.

The following substances do not need to be included in the biennial DEA inventory:

- a) Less than 1 kilogram of any controlled substance (other than a hallucinogenic controlled substance listed in Schedule I)
- b) Less than 20 grams of a hallucinogenic substance listed in Schedule I (other than lysergic acid diethylamide)
- c) Less than 0.5 gram of lysergic acid diethylamide

No inventory is required of known or suspected controlled substances received as evidentiary materials for analysis.

Documentation is required only if a biennial inventory is performed and should be maintained with the annual reference material audit records.

3.4.20 References

ASTM E2327-15 Standard Practice for Quality Assurance of Laboratories Performing Seized-Drug Analysis (2015).

Title 21 Code of Federal Regulations Part 1304 — Records and Reports of Registrants. CFR 1304.11, Am. 2014. Online. https://www.deadiversion.usdoj.gov/21cfr/cfr/1304/1304_11.htm.

Controlled Substances Procedures Manual – Mass Spectrometry; Infrared Spectroscopy

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Michigan Administrative Code, R 338.3101 – R 338.3199q, with specific reference to R338.3151 and R 338.3152

Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG), SWGDRUG Recommendations, v. 7.1 (2016). Online. Available at <http://www.swgdrug.org>.

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3.5 Reagent Reliability

3.5.1 General Information

Reagents are analytical test solutions or substances that are added to a system in order to see whether a reaction occurs.

All reagents are to be prepared from reagent grade chemicals or better.

3.5.2 Verification

Reagents, including stock solutions, shall be tested at the time they are prepared and periodically afterward.

Marquis reagent shall be tested before use or transfer if it has not been tested within 2 months. With the exception of Marquis reagent, all reagents shall be tested before use or transfer to another bottle if they have not been tested within 3 months.

3.5.2.1 Verification Labels

The person testing the reagent shall initial and date the reagent bottle. Whenever the reagent is tested and determined to work properly, the reagent bottle will be initialed and dated by the person testing the reagent.

When a portion of a stock solution is transferred to another bottle for use over a period of time the initials of the analyst making the transfer and the date the solution was most recently tested shall be placed on the bottle containing the transferred portion. Periodically after that time it shall be tested and documented in the manner noted above.

3.5.3 Positive Control(s)

Each bottle of reagent, whether prepared for a single time, a single day, or a period of time, must be verified using one or more standards known to produce a positive response.

Color test reagents shall be verified with at least one standard of a substance that produces a color with the reagent.

Microcrystal reagents shall be specifically verified with at least one standard of a substance that the analyst will use the reagent to later test. A positive result is demonstrated by the presence of characteristic crystals for the substance.

3.5.4 Negative Control(s)

Each bottle of reagent shall be evaluated using one or more standards known to produce a negative response.

For color reagents this would be a substance known to produce no color change.

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For microcrystal reagents a standard shall be used that produces no crystals with the reagent. A negative result is demonstrated by the absence of characteristic crystals the microcrystal reagent will be expected to produce from the substance(s) it will be used to analyze.

3.5.5 Reagent Documentation

Reagent verification shall be documented by maintaining a reagent log (FS-45).

Each bottle of reagent in the unit is to be given a unique identification, which also is placed on the bottle.

The testing of each bottle of reagent shall be documented on a separate page of the reagent log.

The log shall include:

- a) the bottle designation
- b) the initials of the person who tested the reagent
- c) the date the reagent is prepared
- d) the date tested
- e) the drug standard(s) used as a positive control, and positive control test results
- f) the drug standard(s) used as a negative control, and negative control test results.

Additionally, the bottle designations assigned to the control standards when they were verified shall be included in the log.

3.5.6 Reagent Use

When a reagent is used in casework the identification of the bottle and the most recent date of verification shall be written in the case notes.

Including the reagent verification date in the case notes facilitates the review process, as the individual doing the technical review can readily ascertain whether the reagent verification is current.

3.5.7 Reference

IUPAC. Compendium of Chemical Terminology, 2nd ed. (the "Gold Book").

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3.6 Instrument Reliability

In order to ensure that all instruments are operating properly, they must be maintained, tuned, and calibrated. Instrument records should be kept in order to demonstrate that maintenance has been done and the instruments are properly tuned and calibrated. Analysts must be aware of the operating condition of their instruments. Therefore, regular function verification of all instruments is necessary and the following measures will be taken.

3.6.1 Gas Chromatograph/Mass Spectrometers

The tune of all mass spectrometers shall be checked and, if necessary, adjusted on a monthly basis.

An additional tune and calibration are necessary after instrument maintenance and shall be performed by an Equipment Technician.

When checking the tune on the Thermo instruments, a Maintenance Tune shall be attempted prior to the Full Autotune procedure. If the instrument passes the Maintenance tune, no Full Autotune is necessary.

Agilent instruments shall be tuned per manufacturer's specifications.

3.6.1.1 Mass Spectrometer Calibration Check/Maintenance Tune:

Open the DSQ tune program and select "Automatic Tune" from the File drop down menu.

A Maintenance Tune is completed when the "maintenance", "leak check" and "OK" boxes are selected.

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3.6.1.2 Check of MS Tune (PFTBA)

MS methods used in casework are typically based on a standard PFTBA tune.

To ensure a standard PFTBA spectrum tune, the following parameters may be used as a guide when reviewing the S-tune or maintenance tune for use in casework:

Criterion	Value	Range
Mass assignment	m/z 69 219 502	+/- 0.3 amu
Peak shape	Approximately Gaussian	
Counts (m/z 69)	Agilent: 150,000 – 700,000 Thermo: 2.3 – 4 million	
Relative Abundance	m/z 69	100%
	m/z 219	30% – 99%
	m/z 502	1% – 10%
Isotope Ratio	70:69	0.5 – 2
	220:219	3 – 8
	503:502*	5 - 13
Leak Check	Thermo: Passed, <10% reference Agilent: 18:69 <20%, 28:69 <10%	

*NOTE: with a low abundance of m/z 502 this ratio could be less than the range given

3.6.1.3 Full Autotune

An Equipment Technician shall be consulted prior to attempting the Full Autotune.

The Full Autotune function optimizes the tune through the computer. If the instrument successfully passes the Full Autotune, no diagnostic messages are given, and the tune criteria for a Maintenance Tune are met, the instrument is properly tuned.

- a) If the instrument passes but there is a diagnostic message the instrument may be used, but an Equipment Technician shall be contacted.
- b) If the instrument fails to pass the Full Autotune an Equipment Technician shall be contacted to determine whether a service call is necessary or communicate assistance with tuning the instrument. The instrument must be taken out of service until the problem is corrected.

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Out of Service

In the event that the instrument is not functioning properly, the unit supervisor or a designee must be notified and the instrument clearly labeled to indicate that it is out of service (for instance, a sheet of paper with the words "Out of Service" can be placed over the monitor).

After the instrument has been repaired, or the problem was corrected by the Equipment Technician, the label can be removed.

3.6.1.4 Function Verification

On any day the mass spectrometer is used, a function verification sample consisting of approximately 1 mg caffeine and 1 mg cocaine per ml shall be injected into the instrument and the spectra obtained. The volume and concentration of the sample and the gas chromatograph and mass spectrometer operating parameters should be consistent on a day-to-day basis. The spectra obtained will demonstrate that the instrument is producing acceptable spectra and will be an indicator of the sensitivity of the instrument.

The retention times should not change appreciably on a daily basis and the spectra obtained should be of acceptable quality. If the quality of the spectra is not acceptable, the instrument should be tuned and the function verification repeated.

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The following tables show guidelines for caffeine and cocaine m/z relative abundances. An Equipment Technician shall be contacted when these values begin to drift outside of the target ranges.

Caffeine	
m/z	Relative Abundance
55	<40%
67	<40%
82	<40%
109	40-80%
194	100%

Cocaine	
m/z	Relative Abundance
82	100%
94	<50%
96	<50%
105	<50%
182	60-90%
303	8-22%

3.6.1.5 Documentation

Documentation of tune and function verification of gas chromatograph/mass spectrometers shall be retained in a manner which permits review.

Documentation may be either paper or electronic and shall consist of tunes of the instrument and the spectra obtained for its function verification.

3.6.1.6 Maintenance

An Equipment Technician shall perform yearly planned maintenance on all GC-MS instruments in accordance with the FSD Maintained Equipment Maintenance Plan.

Analysts shall perform septum and liner changes as needed, typically monthly. A log shall be maintained of these tasks.

3.6.2 Infrared Spectrophotometers

3.6.2.1 Function Verification

At least once a month and after any maintenance on the instrument a spectrum of polystyrene or a select powder will be recorded.

- a) If the instrument is used in standard modes, polystyrene should be utilized as the standard.

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- b) If the instrument is used in the ATR mode polystyrene and/or a select powder can be selected to be used as the standard.

The spectrum should be an acceptable polystyrene spectrum or select powder spectrum.

The standard spectra should not have changed appreciably from the previous month in quality or signal to noise ratio.

An Equipment Technician must be consulted if a change in the spectrum or the amount of background noise is noticed.

3.6.2.2 Documentation

Hard copies or electronic copies will be kept to demonstrate that the instrument and its internal calibrating system are working properly.

3.6.2.3 Maintenance

An Equipment Technician shall perform yearly planned maintenance on all FTIR instruments in accordance with the FSD Maintained Equipment Maintenance Plan.

Desiccant may be replaced by either an Equipment Technician or an analyst.

3.6.3 Gas Chromatographs

3.6.3.1 Function Verification

The standard (either external or internal) that is run on a case will serve as a function verification of the instrument.

3.6.3.2 Maintenance

An Equipment Technician shall perform yearly planned maintenance on all GC instruments in accordance with the FSD Maintained Equipment Maintenance Plan.

Analysts shall perform septum and liner changes as needed. A log shall be maintained of these tasks.

3.6.4 Balances

Refer to the section on Balances and Weight Measurement in the Controlled Substances Procedures Manual.

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3.6.5 UV-Vis Spectrophotometer

The manufacturer's recommendations for the Perkin Elmer Lambda 25 UV Vis Spectrophotometer for performing a function verification shall be conducted at least once a month and after any maintenance on the instrument.

Hard copies or electronic copies will be kept to demonstrate that the instrument is working properly. There should not be a variance of more than +/- .4 nm in the wavelengths recorded with the Holmium Oxide Filter as the reference standard.

3.6.5.1 UV-Vis Function Validation

The manufacturer's recommendation for function validation shall be conducted by using the following steps:

- 1) Select folder from UVWinlab Explorer, right click on Lambda instrument icon. Select Instrument Performance Verification. Create IPV Setup. Click OK
- 2) Click on New button under Name, enter Holmium
- 3) Check On Demand Only under Timing Box
- 4) Click on Test. Highlight the test IPV Wavelength Accuracy Glass and click on the Add Button. Click on Exit which will prompt the Save the IPV method
- 5) IPV method will state incomplete. Click OK, which will exit the program
- 6) Right click again on the Lambda icon under instruments. Click again on Create IPV Setup. Select Holmium method. Click on the test IPV Wavelength Accuracy Glass. The Lambda instruments are tested with (4) wavelengths (536.2, 459.9, 360.9 and 279.3). Enter the wavelengths for the test. The wavelength accuracy limit is +/- 0.4 nm. Click on exit, program will prompt if any portions are missing. Save method by responding Yes.
- 7) Apply this test to the instrument by right clicking on the instrument icon. Under Instrument Performance Verification select Apply to Instrument. Right click again on the instrument icon and select Perform Now once ready to run the tests.

3.6.6 Discov-IR

The Spectra Analysis GC IR instrument deposits the eluent from a gas chromatograph onto a liquid nitrogen cooled disk allowing for collection of solid phase transmission spectra. The MCT-A detector scans from 4000-650 cm^{-1} and requires cooling with liquid nitrogen prior to analysis.

3.6.6.1 Function Checks

The following checks shall be made and recorded prior to use of the instrument:

- a) Polystyrene shall serve as a monthly function verification prior to use of the instrument
- b) Fill dewars and detector with liquid nitrogen

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- c) Check and fill wash bottles in sampler with appropriate solvent
- d) Check temperatures for transfer line, oven, restrictor, disk and dewar cap
- e) Check and document align and noise

3.6.6.2 Discov-IR Monthly Maintenance

- a) Replace septum, or as needed if more frequently
- b) Clean disk before use as needed
- c) Replace GC inlet O-ring and liner or as needed
- d) Check foreline pump oil level, replenish as needed

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3.7 Comparison with References

Spectral data shall be compared to reference spectra using the following criteria:

3.7.1 Mass Spectral Data

Identification is based upon a full scan mass spectrum.

- a) The unknown spectrum must be compared to a known reference spectrum.
- b) Ions across both spectra shall exhibit general visual correspondence of abundances and nominal m/z values
 - a. The spectra should have the same base peak.
 - b. The molecular ion (when observed) is present with corresponding isotope peaks.
 - c. Some compounds do not produce a molecular ion. In this case, comparison is made based on the fragment ions present.
- c) The presence of extraneous ions in the unknown spectrum should be evaluated for significance, though background subtraction is strongly advised to minimize any background ions from appearing in the sample spectrum.

3.7.2 IR Spectral Data

Identification is based upon a full spectrum.

- a) The spectrum must be compared to a known reference spectrum.
- b) Wavenumbers and relative intensities of peaks present should be in general agreement between the reference spectrum and the unknown.
- c) Extra peaks or missing peaks should be evaluated.

3.7.3 Gas Chromatographic Data

Refer to the procedure for Gas Chromatography.

3.7.4 Reference Spectra

Comparison to a reference spectrum produced from testing of an in-house reference material is the recommended practice for most cases.

For FTIR and GC-IR spectra, comparisons should ideally be made to standard spectra generated with similar instrumental parameters. For example, ATR spectra should be compared to ATR reference spectra.

Comparison to external reference data may be used where an in-house reference material is unavailable.

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3.7.4.1 External References

External reference data shall be shown to be fit for purpose. The veracity of data shall be considered and assessed upon relevant factors including:

- a) Comparability of analytical conditions
- b) Origin of the data
- c) Validation or peer review of the data

The use of external reference data rather than a reference material shall be documented, and where applicable, limitations shall be expressed in the laboratory report.

Refer to Appendix 3.4-A for a listing of sources for external reference data.

3.7.4.2 Documentation of References

Reference spectra used for comparison and identification of a sample FTIR, GC-MS or GC-IR spectrum must be made available for review.

The reference spectrum used for identification purposes can be either

- a) included in the case record object repository
- or
- b) shared from the object repository section objects.

NOTE 1: Library data shared in the object repository section objects file will provide the source of the standard (title, edition and volume).

NOTE 2: If the library data is shared from the object repository section objects the analyst will provide the appropriate information on the instrumental data section of their worksheet which particular reference was utilized for comparison.

All files in the object repositories will be approved by the individual authorizing the reference.

It is the responsibility of the analyst to determine whether or not a particular standard spectrum is suitable for comparison purposes. Skeletal mass spectra references, which lack detail due to an overly dilute standard being used, are generally not acceptable for comparison purposes.

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3.8 (Vacant)

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3.9 Insurance Against Contamination

3.9.1 Redundancy

The Guidelines for Seized Drug Analysis state that multiple tests are to be run on all samples. These tests are to be, at a minimum, one specific structure elucidating test and one selective test.

This redundancy in analysis has two purposes.

- a) To insure against contamination.
- b) To add to the specificity of the entire analysis.

It is a requirement that, when possible, the two tests be run on separate portions of the sample as a further precaution against contamination.

3.9.2 Cleaning of Equipment

All utensils and instruments used in analyses must be carefully cleaned. For example:

Special care must be given to GC syringes in rinsing them a sufficient number of times to insure their cleanliness.

The crystal and anvil of any ATR apparatus must be carefully cleaned between samples.

All glassware, evaporating dishes, FT-IR pellet dies, and laboratory spatulas must be carefully cleaned before each use.

UV cells must be carefully cleaned between samples.

3.9.3 Use of Solvent Blanks

A solvent blank shall be run prior to each case sample run on a gas chromatograph, gas chromatograph-mass spectrometer, or DiscovIR-GC. Additional blanks may be added at the discretion of the analyst.

The solvent blank shall be analyzed with the same instrument operating conditions as the case it precedes. This includes injection parameters and instrument method. This does not include method post-run conditions that occur after the temperature program has completed.

Significant peaks in solvent blank chromatograms shall be investigated and documented in the case record.

Re-run the blank and associated case sample if either of the following has occurred:

- a) a drug, controlled substance, or related compound is detected in any concentration.
- b) an interfering substance is present, such as a compound eluting at the retention time of an analyte in the case.

If the peaks reappear in a blank that has been re-run, the blank should be replaced.

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The instrumental data from the blank shall be kept in the case record with the associated case data.

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3.10 Validation and Verification

3.10.1 Introduction

Validation and verification are quality assurance processes that evaluate a method or equipment to determine if the method or equipment is fit for intended use. Validation or verification is required for all new methods or adaptations of established methods.

3.10.2 Scope

This policy addresses the validation and verification of analytical methods and equipment used in the Controlled Substances Unit. This policy does not replace knowledge, skill, ability, experience, education or training and should be used in conjunction with professional judgment. Safety concerns associated with new methods, if any, are not addressed in this policy.

3.10.3 Significance and Use

All methods, instruments, or technology new to the laboratory are required to be validated or verified, as applicable.

Type of Method	Must be...
Existing Validated Method/Technology (published literature or another laboratory)	Verified by each laboratory
Non-routine validated method	Verified prior to use
New Method	Validated
Change to Method	Validated
New Technology	Validated

3.10.4 General Validation and Verification Requirements

3.10.4.1 Operational Environment

All methods shall be validated or verified in the normal operational environment in which they are expected to be used.

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3.10.4.2 Validation/Verification Plan

All validations and verifications will be planned activities. Plan templates are used to ensure the process adequately covers the validation or verification parameters. Refer to the Appendix for plan templates.

Each validation/verification plan will describe the acceptance criteria for the listed validation/verification parameters.

3.10.4.3 Documentation

The validation/verification process shall be documented and shall include records of:

- a) Personnel involved
- b) Dates
- c) Observations and Data
- d) Conclusions and recommendations
- e) Approval and authorization

Documentation will be maintained in Qualtrax. Historical validation and verification records may be maintained in paper form at each laboratory.

3.10.5 Validation Parameters

All new methods must be validated before use.

3.10.5.1 Qualitative Method Validation

Validation of qualitative methods for the analysis of seized drugs will consider the following parameters:

- a) Specificity/Selectivity
- b) Limit of Detection
- c) Precision (repeatability and reproducibility)
- d) Stability

For qualitative methods with a threshold concentration required to be met for reporting, the validation should also assess:

- e) Linearity
- f) Accuracy at threshold concentration
- g) Precision at threshold concentration

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3.10.5.2 Quantitative Method Validation

Validation of quantitative methods for the analysis of seized drugs will consider the following parameters:

- a) Specificity/Selectivity
- b) Limit of Detection and Limit of Quantitation
- c) Precision
- d) Linearity and working range
- e) Accuracy
- f) Recovery
- g) Measurement uncertainty
- h) Stability

Additional parameters of ruggedness and robustness may also be included in a validation plan.

3.10.6 Verification

Methods that are already validated must be verified before use. This often occurs when equipment is replaced with an equivalent new model or when a method has been validated by one laboratory and is being implemented in a second laboratory.

While a full validation is not required in these cases, the method and equipment must be verified as fit-for-purpose before it can be used for casework.

3.10.6.1 Qualitative Method Verification

Verification of qualitative methods for the analysis of seized drugs will consider the following parameters:

- a) Specificity/Selectivity
- b) Precision (repeatability and reproducibility)

For qualitative methods with a threshold concentration required to be met for reporting, the verification should also assess:

- c) Accuracy at threshold concentration
- d) Precision at threshold concentration

3.10.6.2 Quantitative Method Verification

Verification of quantitative methods for the analysis of seized drugs will consider the following parameters:

- a) Specificity/Selectivity
- b) Limit of Detection
- c) Precision
- d) Accuracy
- e) Measurement uncertainty

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3.11 Appendix

CS-PM App 3 [Abbreviations](#)

CS-PM App 3.3. [Equipment Calibration List](#)

CS-PM App 3.4-A [External References](#)

CS-PM App 3.4-B [Retention of Samples from Casework](#)

CS-PM App 3.10-A [Example GCMS Verification Plan](#)

CS-PM App 3.10-B [Example Qualitative Validation Plan](#)