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ATTENTION: If any portion(s) of the Forensic Toxicology Unit Quality Manual is unclear to any analyst or if a circumstance arises which appears to be outside the scope of this document, it is the responsibility of each analyst to immediately notify the Technical Manager and their Supervisor to seek clarification/approval or obtain guidance on the issue BEFORE proceeding.

1. SCOPE

This manual details the quality assurance program in effect in the Forensic Toxicology Unit (FTU). It is meant to be a source of information for toxicology personnel. This manual should be referred to regularly as a source of information. A system of continuous updating is built into the manual in accordance to OSBI CSD Quality Manual QP 2 – Document Control to allow it to change as laboratory conditions change or as new regulations are disseminated.

Whenever a technician or criminalist is in doubt as to proper procedures in a specific circumstance, the manual should be consulted. Omissions or errors should be immediately reported to the FTU Technical Manager (TM). It is the responsibility of each FTU laboratory employee to ensure that the provisions of this manual are followed. Disagreement with specific requirements or knowledge of changes causing deviation from the procedures should be discussed with the FTU Technical Manager before further work is completed. FTU laboratory personnel are encouraged to comment on the manual and make recommendations for more efficient procedures. The latest revision of this manual is the applicable rule.

2. REFERENCES

The following standards and recommendations guide the requirements set forth in this quality manual. If the reference listed does not include a date, the most recent revision of the referenced document applies.

ISO/IEC 17025:2017

ANAB Accreditation Requirements for Forensic Testing and Calibration (AR 3125)

SOFT/AAFS Forensic Toxicology Guidelines (Version 2006), Society of Forensic Toxicologists Inc. and American Academy of Forensic Sciences, Toxicology Section, 2006.

Organization of Scientific Area Committees (OSAC) for Forensic Toxicology documents

OSBI Criminalistics Services Division Quality Manual (CSD QM-QP)

3. TERMS AND DEFINITIONS

In addition to the terms and definitions listed below, any definition provided in one of the documents listed in section 2 also applies unless the term is defined in this section.

ACCURACY: the closeness of agreement between the value which is accepted, either as a conventional true value or an accepted reference value, and the value found.

ALIQUOT: the portion of the sample used for analysis and is a representative sample of the whole test specimen.

ANALYST: Technician or Criminalist.

ANALYTICAL BATCH: a set of standards, controls, and/or case samples that are contemporaneously prepared and/or analyzed in a particular sequence.

ANALYTICAL SOLUTION: the sample in the form introduced to an instrument. The end result of the sample preparation, extraction, or digestion process.

ANALYTICAL SPIKE: a sample made by spiking an analytical solution after the sample preparation, extraction, or digestion process.

BATCH: samples that are prepared, extracted, or digested together. The samples are analyzed together using the same control samples. Samples in each analytical or extraction batch should be of similar composition.

BIAS: the closeness of agreement between the mean of the results of the measurements of a measurand and the true (or accepted true) value of a measurand. It is reported as a percent difference. The terms accuracy and trueness may also be used to describe bias.

BLANK MATRIX SAMPLE: a biological fluid or tissue (or synthetic substitute) without target analyte(s) or internal standard(s).

CALIBRATION: the set of operations which establish, under specific conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding known values of a measurand.

CALIBRATION MODEL: the mathematical model that demonstrates the relationship between the concentration of analyte and the corresponding instrument response.

CALIBRATION BLANK: usually an organic or aqueous solution that is as free of analyte(s) as possible and prepared with the same volume of chemical reagents used in the preparation of the calibration standards and diluted to the same volume with the same solvent (organic or aqueous) used in the preparation of the calibration standard. Used to give the null reading for the calibration curve.

CALIBRATOR: a solution, either prepared from a reference material or purchased, used to calibrate the assay.

CARRYOVER: the appearance of unintended analyte signal in subsequent samples after the analysis of a positive sample.

CERTIFIED REFERENCE MATERIAL (CRM): a material which specific properties and their values are certified by a technically valid procedure accompanied by, or traceable to, a certificate or other documentation which is issued by a certified body.

COMPETENCY: the demonstration of technical skills and knowledge necessary to perform forensic analysis successfully.

COMPREHENSIVE DRUG SCREEN: a confirmatory test for alkaline or acid/neutral drug identification.

CONTROL CHART: a chart consisting of an expected value (typically the mean) and an acceptable range of occurrences expressed as control limits. The values obtained for measurements versus the date of analysis are plotted to produce the chart.

DECISION POINT: an administratively defined cutoff or concentration that is at or above the method's limit of detection or limit of quantitation and is used to discriminate between positive and negative results.

DILUTION INTEGRITY: the assurance that accuracy and precision are not significantly impacted when a sample is diluted.

DRUG-FACILITATED CRIME (DFC): when an individual is victimized while mentally or physically incapacitated due to the effects of ethanol and/or other drugs.

DRUG-FACILITATED SEXUAL ASSAULT (DFSA): use of a chemical agent to procure sexual contact.

DRUG-FREE BLANK: organic or aqueous solution that is free of drugs.

DUPLICATE SAMPLES: two separate samples taken from the same source.

EDUCATION: formal coursework at an accredited college or university.

EXPERIENCE: direct observation of, and participation in, the practice of a discipline.

FALSE NEGATIVE (FN): a failure to report an analyte that is present above a threshold; or a test result which incorrectly indicates the absence of an analyte.

FALSE POSITIVE (FP): the reporting of an analyte that is not present; or a test result which incorrectly indicates the presence of an analyte.

FLUID: any liquid biological specimen typically pipetted for analysis.

FORTIFIED MATRIX SAMPLE: a blank matrix sample spiked with target analyte(s) and/or internal standard(s) using reference materials.

HUMAN-PERFORMANCE FORENSIC TOXICOLOGY: determines the absence or presence of ethanol and other drugs and chemicals in blood, breath, or other appropriate specimen(s), and evaluates their role in modifying human performance or behavior.

INTERFERENCES: non-targeted analytes (i.e., matrix components, other drugs and metabolites, internal standard(s), impurities) which may impact the ability to detect, identify, or quantitate a targeted analyte.

IONIZATION SUPPRESSION/ENHANCEMENT: a direct or indirect alteration or interference in the instrument response due to the presence of coeluting compounds.

LIMIT OF DETECTION (LOD): the lowest amount of analyte in a sample which can be detected but not necessarily quantitated as an exact value. Also known as analytical sensitivity.

LIMIT OF QUANTITATION (LOQ): lowest concentration at which an analyte can be accurately measured.

MATRIX SPIKE DUPLICATE ANALYSIS: prepared by adding equal and predetermined quantities of stock solutions of a certain analyte(s) to each of two aliquots of a sample prior to extraction/digestion and analysis. Can be used to measure precision.

MATRIX SPIKE SAMPLE: prepared by adding a predetermined quantity of stock solution of representative analyte(s) to actual sample matrix prior to extraction/digestion and analysis. Used to measure accuracy of the method in the sample matrix.

MEASURAND: the quantity intended to be measured.

METHOD BLANK: the calibration blank for methods in which the calibration solutions go through the full sample preparation treatment.

MONITOR: to observe and record activity to measure compliance with a specific standard of performance; routine and ongoing collection of data about the indicator.

NEGATIVE CONTROL (NC): extracted matrix sample that has no reportable response for the target analyte(s) and contains internal standard(s).

NON-STANDARD METHOD: a method not taken from authoritative and validated sources. This includes methods from scientific journals and unpublished laboratory-developed methods.

PER SE LIMIT: a concentration at or above a set value at which a specific conclusion is legally warranted (e.g., 0.08 g/100mL blood ethanol value indicates legal intoxication for a person over 21).

POSITIVE CONTROL (PC): extracted matrix sample that contains a known analyte(s) and internal standard(s).

PRECISION: the reproducibility of the results of quantitative measurements. It is expressed numerically as the coefficient of variation (% CV).

PROFESSIONAL DEVELOPMENT: the education and training that contributes to career advancement and succession planning.

PRIMARY REFERENCE STANDARDS: a standard generally of the highest metrological quality found at a given location, from which measurements made at that location are derived.

QUALIFICATIONS: the combined education, training, and experience of an individual.

QUALITATIVE CONFIRMATION METHOD: an assay designed to identify individual analytes within a sample.

QUALITY ASSURANCE (QA): an integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is the type and quality needed and expected by the client.

QUALITY CONTROL (QC) CHECK: the day-to day operational techniques and activities used by the laboratory to consistently provide accurate analytical results that fulfill the requirements for quality.

QUANTITATIVE METHOD: an assay designed to measure the concentration of an analyte within a sample.

RANGE OF LINEARITY: the interval between the upper and lower concentration (amounts) of analyte in the sample (including these concentrations) for which it has been demonstrated that the analytical procedure has a suitable level of precision, accuracy, and linearity.

REAGENT: a chemical, dilution of a chemical, or combination of chemicals that is employed by the laboratory as specified in a technical procedure.

REAGENT SOLUTION: a liquid or mixture of liquid(s) and chemical(s) prepared in the laboratory for use in an analytical procedure.

REANALYZE: re-pipette/re-extract sample and inject by the correct instrumentation.

REFERENCE MATERIAL (RM): material, sufficiently homogenous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process.

REINJECTION: sample is injected again on the same type of instrumentation. Does not require sample to be re-pipetted or re-extracted.

REPEATABILITY: measurement precision under a set of conditions that includes the same measurement procedure, same operators, same measuring system, same operating conditions,

sample conditions, and same location, and replicate measurements on the same or similar objects over a short period of time.

REPRODUCIBILITY: measurement precision under a set of conditions that includes different locations, operators, measuring system, and replicate measurements on the same or similar objects.

RUGGEDNESS OR ROBUSTNESS: a measure of an analytical procedure's capacity to remain unaffected by small, but deliberate variations in method parameters and provides an indication of its reliability during normal usage.

SAMPLE: laboratory specimen.

SCREENING METHOD: an assay designed to suggest the presence or absence of analytes; thereby indicating further testing may be warranted.

SELECTIVITY: the ability to respond more readily to one target analyte than another.

SENSITIVITY: the ability of a test to correctly identify the presence of a drug. The better the sensitivity, the less false negatives.

SIGNAL-TO-NOISE RATIO: the magnitude of the instrument response to the analyte (signal) relative to the magnitude of the background (noise).

SOLVENT BLANK: made up of a solvent that is introduced to the instrument and has no reportable response for the target analyte(s) or internal standard(s).

SPECIFICITY: the response by the method to a particular analyte sought.

SPIKE: adding a predetermined quantity of analyte(s) to a matrix which is the same or similar to that of the sample of interest prior to sample extraction/digestion and analysis.

STANDARD REFERENCE MATERIAL (SRM): a certified reference material produced by the National Institute for Standards and Technology (NIST) or a certified reference material whose metrological values are approved by a nationally recognized measurement body. NIST is the United States' nationally recognized measurement body.

STABILITY: an analyte's resistance to chemical change in a matrix under specific conditions for given time intervals.

STANDARD OPERATING PROCEDURES (SOP): written analytical instructions that describe how to perform certain organization activities.

TECHNICAL MANAGER (TM): an individual (however named) who is responsible for the technical oversight of the toxicology laboratory.

TECHNICIAN: an individual (however named) who performs basic analytical functions, but does not evaluate data, reach conclusions, or sign a report for court or investigative purposes.

TOXICOLOGIST: an individual (however named) who provides factual information and/or interpretive opinions related to the results of toxicological tests for court or investigative purposes. For purposes of this manual criminalist may also be used.

TRACEABILITY: an unbroken chain of calibrations or comparisons to identified primary standards of the international system (SI) of units of measurement.

TRUE NEGATIVE (TN): a test result which states that no substances are present in the analyzed sample, when they are, in fact, not present at all or are at a concentration less than the cutoff value in the sample.

TRUE POSITIVE (TP): a test result which states that one or more substances are present in the analyzed sample when they are, in fact, present in the sample at a concentration greater than the cutoff value.

UPPER LIMIT OF QUANTITATION (ULOQ): the highest concentration of an analyte in a sample that can be reliably measured with acceptable bias and precision.

VALIDATION, METHOD: the process of establishing the performance characteristics, limitations of a method, and the identification of the influences which may change these characteristics and to what extent.

VERIFICATION: confirmation by examination and provision of evidence that specified requirements have been met. Also interchangeable with the terms, function testing, performance check, and function verification.

WORK PRODUCT: non-retained material that is utilized in testing and/or generated as a function of the analysis process (e.g., alcoholic content dilutions).

WORKING RANGE: the concentration range that can be adequately determined by an instrument, where the instrument provides a useful signal that can be related to the concentration of the analyte.

4. QUALITY MANAGEMENT SYSTEM

4.1 Quality Assurance Program

The FTU's Quality Assurance (QA) program includes all technical and supporting procedures and quality records, which are used to oversee and review the effectiveness of the program. This ensures the FTU adheres to the policies and procedures as described in the Toxicology Quality Manual and Toxicology Standard Operating Procedures Manual.

Through routine unit and discipline meetings, all FTU employees shall be informed of the importance of their activities and how those activities help ensure the FTU meets the objectives of the management system (OSBI CSD Quality Manual 8.0).

4.2 Quality System Records

The FTU will document quality system records in accordance with OSBI CSD Quality Manual QP 2 – Document Control. Quality system records are any logs, worksheets, electronic files, or databases that provide documented support of conformity to the quality management system.

These records include, but are not limited to:

- Method and equipment validation documents;
- Instrument and equipment maintenance and verification records;
- Reagent and chemical logs;
- Training records;
- Proficiency test records;
- Competency test completion records;
- Courtroom testimony monitoring records;
- Laboratory Asset Manager (Chemical inventory) records; and
- Audit records.

Raw instrument data is retained on the hard drive of the instrument computer for the life of the computer in addition to analyzed or derived data stored in the case record. Should a computer be changed out or an instrument removed from service, the last hard drive will be maintained by the TM.

4.3 Reference Journals and Documents

It is common practice to route documents and reference material to members of the unit for review. Each staff member should initial that they have reviewed the material and return it to the FTU Technical Manager. If the document is routed in email form, a response to the email is sufficient. Journals, including the Journal of Analytical Toxicology and Journal of Forensic Sciences, are readily available to all members of the FTU and shall not routinely require initials.

4.4 Impartiality

The FTU will continually evaluate and minimize potential risks to impartiality and conflicts of interest as described in OSBI CSD Quality Manual 4.1.

5. ANALYTICAL/OPERATIONS

The proper selection, collection, and submission of biological and other specimens for toxicological analyses is of paramount importance for scientifically sound interpretation of analytical results. While there are recommended minimum amounts of specific specimens desired to accomplish routine toxicological examinations, specimen amount is often limited. In these cases, the type and amount of specimen submitted may dictate the analyses that are performed.

The analytical process begins when the sample inventorying process begins. The initial step in the process for specimens is the analyte separation. The analytes of interest usually require separation from the biological matrix. After separation, substances are identified. Identification can be grouped into chromatography and immunoassay techniques. Each identification technique, individually, can indicate the presence or absence of a particular analyte. However, for a substance to be reported as positive, a second, more specific technique must be performed. This technique should be based on a different chemical principle whenever possible. At least one technique must utilize mass spectrometry except for ethyl alcohol analysis.

Carrying multiple cases through the analytical scheme together or “batching” is a common and accepted practice. The quality assurance practices established throughout the Forensic Toxicology Standard Operating Procedures allows for this practice while assuring the accuracy, reproducibility, and reliability of analytical results.

5.1 DUI/DUID Case Testing Guidelines

A procedure for the analysis of driving under the influence of alcohol (DUI) or drug (DUID) cases is established to ensure uniformity in testing between criminalists. The procedure applies to all personnel assigned to the FTU.

“Blood Test Officer’s Affidavit” and “Request for Laboratory Examination” (RFLE) forms are provided to submitting agencies. The submitting agency will complete one or both forms when submitting specimens for analysis. These forms request information that will be utilized in case management of DUI/DUID toxicology cases. Table 1 represents the *per se limits* for Oklahoma.

Table 1: Oklahoma *per se limits*

Driver	***Per se limit* (g/100mL)**	**Statute**
Under 21 years of age	Any measurable amount	47 O.S. § 11-906.4; Oklahoma State Dept. of Health Code 310:638-7-4
Aircraft operator	0.040	3 O.S. § 301
Commercial drivers	0.040	47 O.S. § 6-205.2
Other drivers	0.080	47 O.S. § 756
Boating	0.080	63 O.S. § 4210.8

Blood specimens submitted as part of a routine impaired driving investigation will be initially scheduled for an ethyl alcohol analysis, unless ethyl alcohol has already been completed by an accredited forensic laboratory (e.g., Oklahoma City Police Department). If sufficient sample is available, the blood specimens will be scheduled for an immunoassay screen. Samples that were previously tested for ethyl alcohol by an accredited forensic laboratory should be scheduled for an immunoassay screen and not retested for ethyl alcohol unless requested. Biological specimens should be from a living suspect.

Specimens that screen positive by immunoassay analysis, shall be confirmed by a mass spectrometry testing procedure, when feasible, prior to being reported. If drugs not screened for or that were negative by immunoassay are identified during the confirmation procedure, a second confirmation test should be performed and the drug(s) reported. The screening analysis and the confirmation analysis should utilize different chemical principles whenever possible.

If mass spectrometry testing is performed and supports the immunoassay findings, but lacks the criteria for drug confirmation, then a note shall be used to provide clarification and any documentation to support the criminalists' reasoning shall be placed into the case record. If drugs not screened for or that were negative by immunoassay are identified during mass spectrometry testing, but lack the criteria for drug confirmation during the second confirmation procedure, then a note shall be used to provide clarification and any documentation to support the criminalists' reasoning shall be placed in the case record.

If an observation, data, or a test result is rejected, the reason, identity of the person(s) taking the action, and date shall be recorded in the criminalist's notes.

Requests for further analysis may be honored in accordance with OSBI CSD Quality Manual QP 4. The request will be documented in the LIMS system.

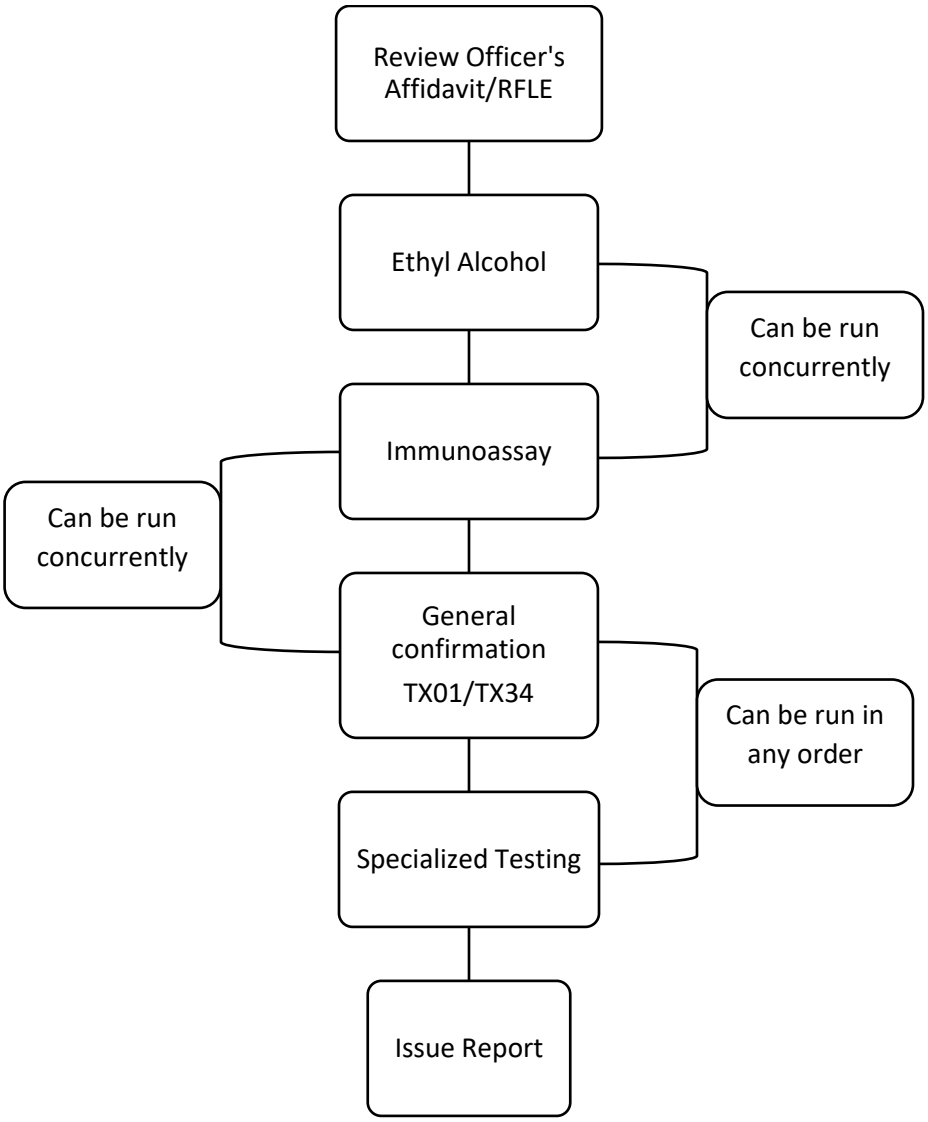
For efficiency purposes, analyses may be completed concurrently (e.g., ethyl alcohol analysis and immunoassay screen; immunoassay screen and TX01 – Basic Drug Screen).

However, an immunoassay screen must be completed successfully prior to completion of any targeted analyses (acid/neutral, cannabinoids, synthetic cannabinoids, benzodiazepines, opiates, and fentanyl and fentanyl analogs). Should one of the concurrent analyses fail, it shall be repeated prior to additional analyses and will still be considered concurrent. Once the ethyl alcohol and immunoassay screen have been completed, mass spectrometry analyses may be completed in any order as long as sufficient sample is available. TX01 or TX34 must be completed on each case unless approved by the FTU TM or designee.

Should any case contain limited sample volume, analyses should be completed one at a time in the typical case flow for DUI/DUID cases in Figure 1 (ethyl alcohol analysis, then immunoassay screen, etc.). Immunoassay screen results must be obtained prior to the beginning confirmation testing (TX01 or TX34).

See typical case flow for DUI/DUID cases in Figure 1.

Figure 1. Typical DUI/DUID and non-DUI/DUID (except ABC) case flow.



5.2 Non-DUI/DUID Cases Testing Guidelines

A procedure for the analysis of non-DUI/DUID cases is established to ensure uniformity in testing between criminalists. The procedure applies to all personnel assigned to the FTU.

RFLE forms are provided to submitting agencies. The submitting agency will complete the form when submitting specimens for analysis. These forms request information that will be utilized in case management of toxicology cases.

Non-DUI/DUID cases include, but are not limited to, drug-facilitated crimes (DFC), alcoholic beverage content (ABC), fatalities that do not involve a motor vehicle, child endangerment,

and poisoning. Drug-facilitated crimes are crimes that are carried out by means of covertly administering a drug to a person with the intention of causing impairment of judgement, motor function, memory, etc. or taking advantage of someone while they are willingly under the influence of ethyl alcohol or another intoxicating substance(s). Biological specimens should be from a living suspect or victim.

5.2.1 Drug-Facilitated Sexual Assault

Drug-Facilitated Sexual Assault (DFSA) is a subset of drug-facilitated crimes. Appropriate tests for any DFSA will be scheduled where the victim alleges blackouts, passing out, dizziness, wooziness, or other symptoms indicative of a DFC. It is recommended that a urine sample be collected from the victim if less than 120 hours have elapsed since the incident. If possible, one hundred (100) milliliters of urine should be collected in a specimen cup and stored refrigerated.

Although most drugs will be undetectable in the blood more than 24 hours after ingestion, blood may prove useful in a DFSA case if collected less than 24 hours after the incident. At least 12 milliliters of blood should be obtained in grey-top test vials containing the preservative sodium fluoride and the anticoagulant potassium oxalate. The blood should be stored refrigerated.

Blood and urine specimens submitted for routine DFSA testing will be initially scheduled for an ethyl alcohol analysis, unless ethyl alcohol has already been completed by an accredited forensic laboratory (e.g., Oklahoma City Police Department). If sufficient sample is available, the blood/urine specimens will be scheduled for an immunoassay screen. Samples that were previously tested for ethyl alcohol by an accredited forensic laboratory should be scheduled for an immunoassay screen and not retested for ethyl alcohol unless requested. Biological specimens should be from a living victim.

Specimens that screen positive by immunoassay analysis, shall be confirmed by a mass spectrometry testing procedure, when feasible, prior to being reported. If drugs not screened for or that were negative by immunoassay are identified during the confirmation procedure, a second confirmation test should be performed and the drug(s) reported. The screening analysis and the confirmation analysis should utilize different chemical principles whenever possible.

If mass spectrometry testing is performed and supports the immunoassay findings, but lacks the criteria for drug confirmation, then a note shall be used to provide clarification and any documentation to support the criminalists' reasoning shall be placed into the case record. If drugs not screened for or that were negative by immunoassay are identified during mass spectrometry testing, but lack the criteria for drug confirmation during the second confirmation procedure, then a note shall be used to provide

clarification and any documentation to support the criminalists' reasoning shall be placed in the case record.

If an observation, data, or a test result is rejected, the reason, identity of the person(s) taking the action, and date shall be recorded in the criminalist's notes.

Requests for further analysis may be honored in accordance with OSBI CSD Quality Manual QP 4. The request will be documented in the LIMS system.

For efficiency purposes, analyses may be completed concurrently (e.g., ethyl alcohol analysis and immunoassay screen; immunoassay screen and TX01 – Basic Drug Screen). However, an immunoassay screen must be completed successfully prior to completion of any targeted analyses (acid/neutral, cannabinoids, synthetic cannabinoids, benzodiazepines, opiates, and fentanyl and fentanyl analogs). Should one of the concurrent analyses fail, it shall be repeated prior to additional analyses and will still be considered concurrent. Once the ethyl alcohol and immunoassay screen have been completed, mass spectrometry analyses may be completed in any order as long as sufficient sample is available. TX01 or TX34 must be completed on each case unless approved by the TM or designee.

Should any case contain limited sample volume, analyses should be completed one at a time in the typical case flow for non-DUI/DUID cases in Figure 1 (ethyl alcohol analysis, then immunoassay screen, etc.). Immunoassay screen results must be obtained prior to the beginning confirmation testing (TX01 or TX34).

5.2.2 Alcoholic Beverage Content (ABC) Cases

An appropriate test(s) for ABC cases will be scheduled when analysis is requested for the identification and confirmation of ethyl alcohol in products currently available for sale, new products that are proposed for sale, or unknown liquids suspected to contain ethyl alcohol. Cases may include, but are not limited to, investigation of minors in possession of alcohol, open intoxicants in vehicle, suspected illegal manufacturing, sale, or distribution of alcohol, and new ethyl alcohol containing products. ABC cases may also include unknown liquids suspected of containing methanol or isopropanol.

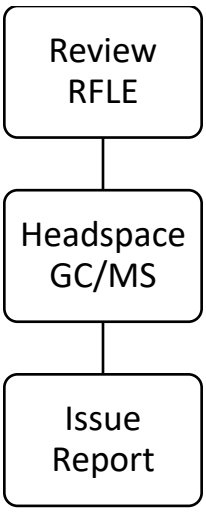
See figure 2 for standard case flow for ABC cases.

5.2.3 Other non-DUI/DUID cases

All other non-DUI/DUID cases that include biological samples will be treated similarly to DFSA cases. Appropriate testing may include: ethyl alcohol analysis, immunoassay, and alkaline drug screen. Any other appropriate test(s) may be scheduled depending on the case circumstances and the officer's initial request.

See figure 1 for standard non-DUI/DUID case flow.

Figure 2. Alcoholic Beverage Content typical case flow.



6. RESOURCE REQUIREMENTS

6.1 Personnel

Personnel who issue a report in the FTU that includes the result of a test, a series of tests, an opinion, or an interpretation, shall meet the minimum education requirements located in the ANAB Accreditation Requirements (AR 3125 Section 6.2.2.1). Personnel must meet requirements as listed in OSBI CSD Quality Manual 6.2.

External and internal proficiency tests will be conducted in accordance with OSBI CSD Quality Manual QP 30 – Proficiency Tests. Proficiency tests will be used to monitor competency of personnel and the quality of the results provided by the criminalist.

6.2 Training

The FTU Technical Manager will maintain a documented training program which meets the requirements outlined in OSBI CSD Quality Manual QP 19 – Training. The FTU Technical Manager shall also oversee training as indicated below:

- Training of new employees as described in OSBI CSD Quality Manual QP 19;
- Retraining of employees as described in OSBI CSD Quality Manual QP 19;
- Training in new areas as described in OSBI CSD Quality Manual QP 21.2 – Evaluation of Methods, Instruments, Equipment, and Software; and
- Continuing education for maintaining skills and expertise in the field of forensic toxicology.

6.3 Facility and Environmental Conditions

The FTU will follow OSBI CSD Quality Manual QP 20 – Laboratory Security to ensure the integrity of tests and evidence. All reference materials and evidence should be stored separately.

The FTU does not have any specific requirements for environmental conditions which could impact results as described in 6.3.2 in the OSBI CSD Quality Manual.

6.4 Reference Collections

Reference collections of data encountered in casework are maintained for identification and comparison or interpretation purposes (e.g., mass spectra). There is no requirement for measurement traceability of reference collection items/materials. However, reference collections must be "traceable". The "history" of each item must be known and documented.

Purchased data libraries (reference collections) are fully documented and uniquely identified. No changes will be made to purchased reference collections. Examples of such libraries include GC/MS NIST, Wiley, and Pfleger.

Data libraries obtained from reputable forensic sources are fully documented and uniquely identified. No changes will be made to these reference collections. The addition or removal of forensic libraries must be approved by the FTU Technical Manager. The OSBI toxicology libraries are the only libraries approved for reporting results.

For in-house libraries (i.e., GC/MS OSBI_Toxvxx.L and OSBI-100_Toxvxx.L) each entry is automatically identified by a unique tracking number generated by the instrument software. These libraries will be generated or modified by the FTU Technical Manager or designee.

When updating in-house libraries, prepare an approximate 2 µg/mL solution of the CRM and analyze by the appropriate qualitative instrument method. If the solution is too concentrated or too dilute, a different concentration may be used to achieve good chromatography.

NOTE: chloroform works well as a diluent for GC/MS.

Verify the CRM for peak purity and check the spectrum against an outside spectral source such as a journal or validated library and/or the Certificate of Analysis (COA). If the CRM is confirmed for use, then the CRM may be placed into the library.

If a CRM is not available, a reference material (RM)(standard) can be used instead for qualitative purposes only. Prior to use, the RM should be analyzed by GC/MS and the

spectrum checked against an outside spectral source such as those listed above. If the RM is confirmed for use, then the RM may be placed into the library.

Recommended steps for adding a new entry to the in-house library once it has been determined to be acceptable:

1) In data analysis, select peak of compound to be added
2) Select “spectrum”
3) Select “edit library”
4) Select “add new entry”
5) Complete form
    a. Include the following information in the “name” box:
        i. Compound name in all caps
        ii. Retention index
        iii. Drug standard identifier (vendor and lot number)
        iv. Initials of person entering the data
        v. Date
        vi. Column type in parenthesis

        (e.g., FLUBROMAZOLAM RI:3085 Cayman L#0464453 TLA 08-17-17 (100))
    b. Compound name in “Miscellaneous Information” box
    c. Molecular formula
    d. CAS number (if available)
    e. Retention index (in “User Index” box)
    f. Select “include in search” box
6) Add the retention index for the 5% and 100% columns in “OSBI_Tox Library” Spreadsheet in the “Toxicology_Lab” folder on the server.
7) Print to pdf and store with outside spectral source used for comparison in the “Toxicology_Lab” folder on the server.

6.5 Equipment

Equipment used in the FTU that requires calibration, shall be calibrated by appropriately accredited calibration service suppliers per CSD Quality Manual QP 23. The calibration certificate should contain the uncertainty of measurement for the calibration and be NIST traceable. Copies of calibration certificates generated by the vendor, certificate of

accreditation for the vendor, and the scope of accreditation for the calibration year will be stored electronically in the "Toxicology_Lab" folder on the server.

6.5.1 Calibration of Analytical Equipment

When practical, analytical equipment (i.e., balances, pipettes, and volumetric flasks) requiring calibration shall be labeled, coded, or otherwise identified to indicate the status of calibration.

The label or tag found on or near the equipment contains the following information:

- Unique identification number;
- Date of last calibration; and
- Date of the next calibration (if applicable)

Equipment that is scheduled to be calibrated is tagged as above. Volumetric flasks may be labeled in a way that prevents the loss of the above information as long as the above required information is available for review. This information may be stored in the "Toxicology_Lab" folder on the server.

6.5.2 Pipettes

All pipettes, pipette diluters, automatic diluters, dispensettes, and syringes used for the preparation of calibrator solutions that require measurement traceability or sample preparation (i.e., steps that affect overall measurement uncertainty) should be calibrated at least annually by an appropriately accredited calibration service supplier. Autosampler syringes used for sample introduction to analytical instrumentation do not require calibration. All calibrated pipettes shall be uniquely identified and appropriately labeled as delineated in Calibration of Analytical Equipment.

Verification checks are needed to maintain confidence in the calibration status of a pipette between calibrations. After pipettes have been externally calibrated and returned to the OSBI FTU, an acceptable verification check must be completed before returning pipettes to service. If pipettes are calibrated by an external vendor on-site at the OSBI FSC, no verification check is needed since the pipettes remained in the custody of the laboratory. Approximately six months between external calibrations, an acceptable verification check will be completed before continuing use of each pipette.

The verification check will consist of evaluating each pipette to ensure that it is within tolerance. For adjustable volume pipettes, the same volumes used by the calibration company should be verified.

<u>Pipette Verification using a Balance</u>

Five measurements are required to confirm accuracy. One weigh boat may be used for multiple readings with a tare performed between each weight measurement. Multiple pipette calibration checks will require the boat to be emptied and a tare performed. The pipette precision must be within ± 5% of the coefficient of variation (CV) and the accuracy must be within ± 5% to be acceptable for use. For small volume pipettes (less than 20 µL), pipettes must be within ± 10% CV and ± 10% accuracy to be acceptable for use. This will be performed gravimetrically with water and documented on a spreadsheet/pdf stored electronically in the "Toxicology_Lab" folder on the server.

Should the pipette fail the verification check, the FTU Technical Manager shall be notified and will determine the next course of action which may include removing the pipette from service and/or sending it to an authorized vendor for repair. If a pipette appears to be out of calibration between verification checks, a verification check may be completed. If the pipette fails the verification check, then the FTU Technical Manager will be notified and will determine the next course of action which may include removing the pipette from service and/or sending it to an authorized vendor for repair.

All other pipettes not used for traceability will have a verification check completed concurrent with the verification checks for calibrated pipettes.

As needed, the pipettes can be cleaned with a 1:10 bleach:water solution. When maintenance has occurred, a copy of the maintenance repair documentation will be stored electronically in the "Toxicology_Lab" folder on the server.

Calculations:

Average: $\bar{x} = (x_1 + x_2 + x_3 + \cdots + x_n) \,/\, n$

Standard Deviation (weight): $s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n}}$

Precision Error$_{weight}$ (%CV): $\%CV = \frac{s}{\bar{x}} \times 100$

Relative Standard Deviation (RSD): $RSD = \frac{s}{\bar{x}}$

% Relative Standard Deviation: %RSD = RSD*100

Accuracy Error$_{volume}$ (%): $\% = \frac{\overline{x}_{Volume}}{Pipette\ Volume}$*100

6.5.3 Volumetric Glassware

Calibrated class A volumetric glassware shall be used for the preparation of calibrators. The calibration shall be completed by an accredited calibration service supplier prior to use. Volumetric glassware used in the preparation of calibrators should be dedicated for this purpose and maintained and stored as to protect its integrity. After initial calibration, scheduled recalibration shall recur at least once every ten (10) years by an appropriately accredited calibration service provider.

6.5.4 Balances

Balances shall be calibrated annually by an appropriately accredited calibration service supplier. Internal calibration is not used.

6.5.5 Digital Thermometers

Thermometers used to verify proper storage of certified reference materials shall be calibrated at least every two (2) years by an appropriately accredited calibration service supplier or NIST certified digital thermometers can be used. The NIST certified digital thermometers should be replaced according to the manufacturer's recommendations. Batteries can be replaced without affecting the calibration.

Other thermometers that do not affect the accuracy and validity of the test results do not require calibration.

The Digital Thermometer Assignments, for calibrated digital thermometers, will be logged and maintained on a spreadsheet found in the "Toxicology_Lab" folder on the server.

6.5.6 Reference Standards

Reference standards (e.g., mass reference standards) shall be calibrated at least annually by an appropriately accredited calibration service supplier.

The certified mass reference standard will be kept in storage containers that will protect them from contamination and damage. They will be transported in the respective storage containers and not in buckets and/or boxes.

The mass reference standards may be used to verify the balance is working correctly prior to a pipette verification check. If, at any time, a mass reference standard is outside of the acceptable 1% range on the balance, the mass reference standard will be weighed on a second balance to determine if the issue is with the balance and not the mass reference standard. If it is determined the issue is with the mass reference standard, it will be sent to an outside vendor for re-calibration and re-certification before it can be used again. The outside vendor must maintain a quality system that meets or exceeds

the requirements set forth in ISO/IEC 17025. If it is determined the issue is with the balance, the balance will be re-calibrated and re-certified before it can be used again. The vendor must maintain a quality system that meets or exceeds the requirements set forth in ISO/IEC 17025.

6.5.7 Certified Reference Materials

If traceability of a measurement will be established through a reference material, the FTU shall establish it through the use of one or more certified reference material(s) (CRM). If available, the CRM shall be obtained by the FTU from a supplier that meets the requirements outlined in OSBI CSD Quality Manual QP 26 – Reference Materials.

If a CRM is not available to the FTU from a supplier that meets the criteria in OSBI CSD Quality Manual QP 26, then the FTU shall perform an evaluation of the CRM supplier. This evaluation shall ensure the CRM supplier meets competency, measurement traceability, and measurement capability requirements in ISO/IEC 17025 or in the ILAC Policy on the Traceability of Measurement Results. The FTU shall keep objective evidence of this evaluation.

Reference materials will be handled as required by OSBI CSD Quality Manual QP 26. Vendor evaluation must be performed before a CRM can be used.

6.5.7.1 Purchasing

When purchasing a standard, the following should be completed:

- Confirmation of accreditation requirements of the CRM vendor;
- Confirmation of the CRM vendor's capability;
- Obtain the scope of accreditation either from the vendor, the vendor's website, or from the accrediting body;
    - From the accrediting body's website: locate the CRM supplier in the list of accredited laboratories and download/print the scope of accreditation; and
- Review the scope of accreditation for the service(s) required and retain a copy of the scope of accreditation as a record of vendor evaluation.

6.5.7.2 Labeling and Storage

When a reference material is received, a Certificate of Analysis (COA) for that reference material should be obtained when available.

After the data on the COA has been reviewed, the COA will then be marked received (i.e., “rec’d” or equivalent notation), initialed and dated indicating the standard is ready for use.

Reference material COAs will be stored electronically in the appropriate folder in the “Toxicology_Lab” folder on the server or in Chemical Inventory. If the COA is stored on the server, the COA should be moved to the “0-Archived” folder in the “Toxicology_Lab” folder on the server, when it has been consumed or expended. If the COA is stored in Chemical Inventory, then the CRM should be moved to the expended list after being consumed.

A listing of reference materials in the laboratory is documented electronically on the “Toxicology Drug Standards” form found in the “Toxicology_Lab” folder on the server. The list is to be updated as reference materials are added to/expended from the inventory.

The reference material list will contain, at a minimum, the chemical name or description, source, manufacturer’s lot number, the laboratory lot number (if the manufacturer’s lot number is not used), storage location, and expiration date, if present.

Reference materials will be labeled with date of receipt and recipient’s initials. When opened, it will be labeled with the date opened and the opener’s initials.

Any reference material remaining that cannot be resealed (e.g., ampoules) will be transferred into another storage vial and labeled with all of the above-mentioned information.

Verification of the reference material by Gas Chromatograph/Mass Spectrometer (GC/MS) or Liquid Chromatograph tandem Mass Spectrometer (LC/MS-MS) is not required if a COA has been obtained. Otherwise, verification by GC/MS or LC/MS-MS must be done prior to the reference material being used in casework.

The receipt and usage of all solid dosage drug standards (10 mg) and liquid standards (10mg/mL or above) will be accounted for on a “Drug Receipt and Usage Log” form. The log form should be completed as thoroughly as possible upon receipt of such a drug reference material. All “Drug Receipt and Usage Log” forms will be maintained in the “Toxicology_Lab” folder on the server. When the drug reference material is depleted, the “Drug Receipt and Usage Log” form will be zeroed out in the “Amount Remaining” column and the form moved to an archived folder in the “Toxicology_Lab” folder on the server. Reference materials that need to be destroyed are to be zeroed out and a notation made on the “Drug Receipt and Usage Log” that the reference material has been destroyed. The form

will then be archived in the “Toxicology_Lab” folder on the server. A drug destruction inventory form will be filled out and submitted to the Physical Evidence Supervisor per O.S. § 63-2-315 when the drug is submitted for destruction.

When a reference standard is transferred to a storage container, the container should be labeled with the name of the standard, the manufacturer, the manufacturer’s lot number, the date the standard was received, the initials of the receiving analyst, the date the standard was opened, the initials of the analyst opening the standard, and the expiration date, if applicable.

Secondary standards stored in a container, should be labeled with the standard name, the assigned OSBI lot number, if applicable, the initials of the analyst that made the secondary standard, and the date made.

Upon receipt of a federally controlled Schedule I or II substance (either in powder or as a dilute solution) the designated Criminalistics Administrator must be notified per DEA requirements.

6.5.8 Modifications to Reference Materials

If a reference material used to establish measurement traceability is diluted, such as a stock or working solution, then the equipment used should be calibrated as delineated previously. This applies even if the reference material is not certified.

6.6 Other Equipment

General laboratory equipment used during sample preparation (i.e., centrifuges, rotators, shakers, water baths, evaporators, extraction manifolds, and heating blocks), that does not significantly affect the accuracy and validity of the test result, does not require calibration to establish measurement traceability. However, calibration or verification as a maintenance procedure may be used to ensure proper functioning of the equipment.

6.6.1 Software Evaluation

Commercial, off-the-shelf software (i.e., word-processing, database, and statistical programs) used within its designated application range may be considered to be sufficiently validated.

6.6.2 Centrifuges

All recommended centrifuge speeds are approximate in order to achieve the appropriate separation of layers. Therefore, no intermediate tachometer checks to verify the speed of the rotor are required.

Clean the centrifuge as needed.

If a centrifuge is taken off-line for repair/maintenance, it should be labeled as out of service until it is repaired. Follow requirements in OSBI CSD Quality Manual 6.4.

6.6.3 Refrigerator

Due to the nature of biological evidence, specimens being retained for analysis should be kept refrigerated when not being analyzed. To ensure these specimens are maintained within appropriate refrigeration temperatures, Physical Evidence Quality Procedures Manual PE QP 2.4 – Evidence Refrigerator and Freezer Maintenance, shall be followed for maintenance and monitoring refrigeration.

The refrigerator temperatures shall be recorded weekly with the same information included on Physical Evidence Quality Procedures Manual PE QPA 2.4.1.

When a refrigerator or freezer temperature falls outside the acceptable range, the following actions shall be taken to investigate and correct the issue:

- The unit shall be inspected to attempt to determine the cause of the variance. If the cause is readily identified (e.g., a door not closed properly, evidence stacked in a fashion that prevents air circulation, etc.), then appropriate steps will be taken to correct the issue and the temperature will be closely monitored to ensure that the steps taken corrected the problem.
- If the cause cannot be easily determined by in-house personnel, the unit shall be emptied. The contents shall be transferred to a working unit (if possible) or to temporary storage until expedient arrangements can be made for proper storage. The unit shall be marked with an “Out of Service” sign. The date shall be recorded in the refrigerator log. The FTU Supervisor assigned to refrigerators, or designee, shall arrange to have the unit repaired or replaced. Follow requirements in OSBI CSD Quality Manual 6.4.

6.6.4 Water Baths

Thermometers used for water bath temperature checks do not need to be NIST traceable since the heat of the water bath for the nitrogen evaporator is used to help speed up the drying process and does not affect the measurement traceability.

6.6.5 Hydrogen Generators

The deionizer bags should be replaced every six months. Desiccant cartridges should be replaced when needed. This is indicated by color change of the desiccant. Any maintenance performed shall be documented on the maintenance log.

6.7 Laboratory Instrumentation

All equipment in the FTU is sufficient for the intended purpose, and should be kept in a state of maintenance and calibration consistent with its use. Instrument manuals are located in the instrument room, the ELISA room, on the instrument's computer desktop, or in the "Toxicology_Lab" folder on the server. Manufacturer suggested maintenance can be completed as needed or suggested if not required by the FTU Quality Manual. Directions for instrument use are located in the "Toxicology_Lab" folder on the server.

Should an instrument function outside acceptable limits and is unable to be corrected as soon as feasible, the instrument should be placed out of service and the requirements in OSBI CSD Quality Manual 6.4 shall be followed and the FTU Technical Manager and/or Supervisors or designee should be notified.

6.7.1 New Instrument Installation

1) Obtain documentation that demonstrates that the instrument performs to the manufacturer's specification from the Instrument Service Engineer.
2) Verify appropriate software is loaded and test functionality.
3) Perform instrument self-check, as needed.
4) Run check solutions, positive controls, or calibrators for appropriate instrumentation to demonstrate the instrument is fit for use (i.e., appropriate sensitivity, specificity, accuracy, precision, chromatography, or identification of the components of a mixture).
5) Backup methods and data analysis macros to writable CD/ROM or other suitable long-term storage media.
6) Retain instrument verification documentation in the new instrument log book or electronically.
7) A summary of the verification shall be forwarded to the FTU Technical Manager for approval prior to placing the new instrument into service.
8) If the instrument does not meet expectations or acceptance criteria, label it as "Out of service" and notify the FTU Technical Manager as soon as possible so the issue can be resolved.

6.7.2 Equipment Identification and Records

Per OSBI CSD Quality Manual QP 24 – Calibration and Handling of Equipment and OSBI Policy 209 – Asset and Personal Issue Inventory, all appropriate equipment in the OSBI equipment inventory system is labeled with a unique identification number (e.g., OSBI bar code number).

The FTU maintains an inventory of its major equipment in the OSBI-FSC inventory system database.

6.7.3 Tecan ELISA System

The Tecan ELISA system will be handled in accordance to FTU SOP Manual TX04 ELISA Drug Screen. Additionally, Direct Assay ELISA kits will be labeled with a unique identifying number when received. The lot number for the kit can be used as the unique identifying number. When a kit is opened, the bottles of TMB substrate, stop solution, conjugate, and synthetic urine will be labeled with this unique identifying number before they are removed from the kit.

Prior to batch analysis, ensure the water containers are filled.

After analysis, remove any remaining stop and TMB solutions from the instrument deck and return to the refrigerator. Cap all conjugate solutions and return to the refrigerator. Remove specimens and controls from the instrument deck.

Preventative maintenance (PM) should be completed by the manufacturer when included in the instrument maintenance contract.

A monthly rinse should be completed as recommended by the manufacturer. The instrument will prompt the user through the process. This should be documented on the maintenance log.

If part of the instrument is sent out for repair, it must be shown to function properly before being placed back in service. This can be done by reviewing the low positive control, high positive control, and negative control data before reviewing sample data.

Any maintenance performed shall be documented on the appropriate instrument log with the same information included in OSBI CSD Quality Manual QP 24 – Calibration and Handling of Equipment.

6.7.4 Headspace Gas Chromatographs (Mass Spectrometer)

Most toxicology procedures are performed in a batch and therefore most maintenance procedures are performed prior to running a batch of samples.

Any maintenance performed shall be documented on the appropriate instrument log with the same information included in OSBI CSD Quality Manual QP 24 – Calibration and Handling of Equipment.

Autotune and Tune Evaluations should be completed on all headspace GC/MS instruments as described in section Autotune and Tune Evaluation.

6.7.5 Liquid Chromatograph Mass Spectrometers

Most toxicology procedures are performed in batches and, therefore, most maintenance procedures are performed prior to running a batch of samples.

Any maintenance performed shall be documented on the appropriate instrument log with the same information included in OSBI CSD Quality Manual QP 24 – Calibration and Handling of Equipment.

- As Needed – Fill mobile phase and rinse solution bottles (does not need to be documented on the maintenance log).
- Weekly – Ballasting procedure (if applicable) and isopropanol rinse.
- Yearly – PM completed by manufacturer when included in instrument maintenance contract.

6.7.6 Gas Chromatograph Mass Spectrometers

Most toxicology procedures are performed in batches and, therefore, most maintenance procedures are performed prior to running a batch of samples.

Any maintenance performed shall be documented on the appropriate instrument log with the same information included in OSBI CSD Quality Manual QP 24 – Calibration and Handling of Equipment.

A solution of each n-alkane from C10 to C30, C32, C34, and C36, also known as a hydrocarbon ladder, will be injected for any of the following: prior to first day of use of the month, prior to use of a new column for the GC/MS, and every time the column is cut.

There shall be separation of each n-alkane including defined, symmetrical peaks. Only components of the hydrocarbon ladder should be present. If additional peaks are observed that either coelute with a n-alkane or have a peak height that is more than 25% of the peak height of the lowest n-alkane, the hydrocarbon ladder should be reinjected or reanalyzed. For example, if the n-alkane with the lowest peak height is at a height of 100 units, then any additional peaks cannot exceed 25 units.

If additional peaks are still observed that do not pass the above criteria, then the analyst shall manually scan these peaks to determine if they are junk peaks. If the peaks are determined to be junk peaks and are not integrated or coeluting with an n-alkane, the hydrocarbon ladder may be used with a note, on the hydrocarbon ladder document, stating that the peaks have been manually scanned and should include the date and the analyst's initials. If the peaks coelute with an n-alkane or are not found to be junk peaks,

maintenance is recommended (see Maintenance section). If the issue is not remedied by maintenance, the FTU Technical Manager or designee will be notified.

If these criteria are not met, then the hydrocarbon ladder must be repeated. Casework will not by analyzed prior to an acceptable hydrocarbon ladder being performed.

6.7.6.1 Autotune and Tune Evaluation

An autotune and tune evaluation will be performed on the GC/MS daily (prior to analyzing the first sample sequence) or after the completion of an overnight analysis. An autotune and tune evaluation should also be completed after column maintenance is completed on the instrument. The autotune and tune evaluation are not required to be, but can be, listed on the maintenance log. Exceptions to these requirements should have prior approval of the FTU Technical Manager.

If the instrument stops during analysis due to an autosampler error (e.g., dropped vial, bent syringe, etc.) or a software error interrupting a sequence there is no need to autotune the instrument and does not require approval from the FTU Technical Manager, provided that the instrument has not been idle longer than 15 hours.

1) Perform the autotune (atune) with perfluorotributylamine (PFTBA) as the tuning solution.
2) Compare the autotune report to previous ones and notify the FTU Technical Manager or designee of any major variations.
3) The mass assignments of the three tuning masses in the upper portion of the report shall be within ± 0.2 amu of 69.00, 219.00, and 502.00. If the deviation is larger than ± 0.2 amu, document the deviation on the appropriate maintenance log. Perform another autotune. If the problem persists, perform maintenance as needed and document the maintenance and deviation on the maintenance log. If the problem cannot be solved through maintenance, notify the FTU Technical Manager or designee. The instrument shall remain out of service until the problem is corrected. Follow requirements in OSBI CSD Quality Manual 6.4
4) The peak widths (Pw50) of the three tuning masses shall be 0.60 ± 0.10 amu and the peaks should generally be smooth and symmetrical. If the deviation is greater than 0.10 amu, document the deviation on the appropriate maintenance log. Perform another autotune. If the problem persists, perform maintenance as needed and document the maintenance and deviation on the maintenance log. If the problem cannot be solved through maintenance, notify the FTU Technical Manager or designee. The

instrument shall remain out of service until the problem is corrected. Follow requirements in OSBI CSD Quality Manual 6.4.

5) The 70/69 isotopic ratio shall be from 0.5 – 1.6, the 220/219 ratio shall be from 3.2 – 5.4, and the 503/502 ratio shall be from 7.9 – 12.3. If these requirements are not met, document the deviation on the appropriate maintenance log. Perform another autotune. If the problem persists, perform maintenance as needed and document the maintenance and deviation on the maintenance log. If the problem cannot be solved through maintenance, notify the FTU Technical Manager or designee. The instrument shall remain out of service until the problem is corrected. Follow requirements in OSBI CSD Quality Manual 6.4.

6) The abundance of the 18 amu peak compared to the abundance of the 69 amu peak shall not be greater than 20%. The abundance of the 28 amu peak compared to the abundance of the 69 amu peak shall not be greater than 10%.

   NOTE: Background noise often increases if the ratio of abundance of the 18:69 amu peaks is above 10%.

   If these requirements are not met, document the deviation on the appropriate maintenance log. Perform another autotune. If the problem persists, perform maintenance as needed and document the maintenance and deviation on the maintenance log. If the problem cannot be solved through maintenance, notify the FTU Technical Manager or designee. The instrument shall remain out of service until the problem is corrected. Follow requirements in OSBI CSD Quality Manual 6.4.

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

8) If an air leak is detected, the air leak shall be isolated and corrected and the autotune repeated. If the problem persists, document the deviation on the appropriate maintenance log and notify the FTU Technical Manager or designee. The instrument shall remain out of service until the problem is corrected. Follow requirements in OSBI CSD Quality Manual 6.4.

9) Autotunes and tune evaluations will be stored in the “Toxicology_Lab” folder on the server and organized by instrument name and year. The Autotune will be saved in the following format: MMDDYYinitials (e.g., 080825MNB). The tune evaluations will be saved in the following format: MMDDYYTEinitials (e.g., 080825TEMNB). These documents will be attached to the appropriate batch run which is stored indefinitely in the image vault.

6.7.6.2 Maintenance

A routine maintenance schedule is a suggested minimum guideline. The maintenance schedule will be determined by the instrument operator based upon instrument use and performance.

- Wash vials
    - Rinse and/or fill with the appropriate solvent as needed, when in use.
    - Post-maintenance check: none
    - Does not need to be documented on the maintenance log.
- Liner
    - Inspect chromatography and replace as needed.
    - Post-maintenance check: none
- Syringe
    - Inspect periodically for cleanliness and ease of motion. Replace as needed.
    - Post-maintenance check: none
- Pump oil
    - Change every six months or per PM contract (if applicable).
    - Post-maintenance check: successful autotune
- Clean source
    - Clean as needed.
    - Post-maintenance check: successful autotune
- Gold seal
    - Replace as needed.
    - Post-maintenance check: successful autotune
- Septa/Merlin Microseal
    - Replace as needed.
    - Post-maintenance check: successful autotune

Any maintenance performed shall be documented on the appropriate instrument log with the same information included in OSBI CSD Quality Manual QP 24 – Calibration and Handling of Equipment unless otherwise noted above.

6.8 Measurement Traceability

For equipment and certified reference materials used to establish and maintain measurement traceability, proper handling and storage procedures which meet or exceed manufacturer's recommendations must be followed.

The FTU uses the procedure outlined in OSBI CSD Quality Manual QP 23 – Measurement Traceability to ensure adequate traceability of measurements that impact measurement uncertainty.

OSBI CSD Quality Manual QP 24 – Calibration and Handling of Equipment will be used to ensure safe handling, transport, storage, use, and maintenance of measuring equipment.

6.9 Purchasing Services and Supplies

The FTU follows the purchasing of supplies as specified by OSBI CSD Quality Manual 6.6.

Order only sufficient volumes of chemicals to be consumed prior to the established expiration date. This will reduce the need and expense of disposal of unused/expired chemicals.

All chemicals must be marked with the date received, initials of the receiver, and expiration date, if applicable. Additionally, when the chemical is opened, the date opened and initials of the opener must be added.

Upon receipt in the FTU, supplies will be checked against the packing slip to ensure the correct item(s) was received. If a substitution is necessary, the FTU Technical Manager should be consulted to determine if the substitution is acceptable per OSBI CSD Quality Manual QP 8.1 – Ordering, Receiving, and Verifying Reagents and Supplies.

6.10 Reagents and Consumable Supplies

6.10.1 Reagents

A reagent is broadly defined as a chemical, dilution of a chemical, or combination of chemicals that is employed by the laboratory as specified in a technical procedure.

> **NOTE**: Reagent is not synonymous with reference standards or reference materials which are used directly to (or in prepared standards that) calibrate, provide qualitative identification, or verify quantitative accuracy.

Chemicals used in qualitative and quantitative testing should be of at least American Chemical Society (ACS) reagent grade or better.

Solvents used in the toxicology section shall be high quality, low residue solvents (e.g., HPLC grad Omnisolv, Optima, etc.).

Prepared reagents or reagents transferred to smaller containers will be labeled, at a minimum, with the name of the reagent, lot number, and expiration date, if applicable.

Solvents that have a COA (or similar document) do not need further function verification. The COA shall, if available, be maintained in Chemical Inventory.

All laboratory prepared reagents shall demonstrate proper function. Reagents and supplies identified as affecting the quality of analysis must also demonstrate proper function. Function verification should include testing the reagent in the same manner it will be used in casework. Prepared reagents are checked for reliability after preparation by evaluation of appropriate blanks, negative and positive controls, and/or positive calibrators.

Function verification may be performed either prior to the reagent's use in casework or concurrent with casework as long as there is enough sample to reanalyze if necessary. Attachment of the appropriate controls to the reagent in Chemical Inventory is acceptable proof of verification. If a reagent is used for both controls and samples, only the negative control data needs to be attached in Chemical Inventory.

A list of critical reagents should be stored in the "Toxicology_Lab" folder on the server. All critical reagents that are prepared in-house will demonstrate proper function as described above. For purchased critical reagents, new brands and/or grades will be function tested even if a COA is received. If the purchased critical reagent is the same brand and grade as a previously function tested lot and has a COA, additional function testing is not required.

In accordance with OSBI CSD Quality Manual QP 8.1 – Ordering, Receiving, and Verifying Reagents and Supplies, the necessary information will be retained in Chemical Inventory.

Examples of reagents may include the following:

- A dry chemical such as the salt, sodium chloride
- A liquid chemical such as the solvent, ethyl acetate
- A liquid chemical prepared from either a dry or liquid chemical such as a buffer or dilute acid (e.g., sodium phosphate buffer or 0.1 M acetic acid)
- A pre-diluted or pre-mixed chemical specified for use on a particular piece of equipment or by the manufacturer of a particular system (e.g., a labeled antibody or antigen complex used for immunoassay testing).

6.10.2 Consumable Supplies

A consumable supply is any material other than a reagent which is purchased for laboratory use. It is not equipment or instrumentation, but may be a component of either (e.g., a GC injector inlet liner is used on the instrument as a consumable supply).

Any consumable supply that has been defined as critical, will be purchased exclusively from an approved supplier as described in OSBI CSD Quality Manual QP 9 – Evaluation of

Suppliers. A list of critical supplies will be included with the approved suppliers list in the "Toxicology_Lab" folder on the server.

6.10.3 Chemical Storage

Chemical storage will be in accordance with OSBI CSD Quality Manual QP 8.1 – Ordering, Receiving, and Verifying Reagents and Supplies.

6.10.4 Determination of Expiration/Discard Dates

For re-verifications, the paperwork should be included in Chemical Inventory for the first use after the expiration/discard date and each consecutive year. For example, if the chemical expires 02/22, the first test after expiration is completed on 03/01/22, this data will be stored in Chemical Inventory and data from the first run after 03/01/23 will also need to be added to Chemical Inventory if the chemical is not discarded before then, and so forth until the chemical is discarded. The re-verification/expiration due date should be placed on the container.

6.10.4.1 Commercially Available Chemicals

Chemicals that don't have an expiration or discard date will be continually evaluated when analyzing controls in the test using those chemicals. When commercial chemicals and reagents have an expiration or discard date, then they will be re-evaluated when they expire to determine if they continue to be "fit for purpose" or discarded.

6.10.4.2 Reagent Solutions

Reagent solutions should expire or be discarded, one (1) year from the date of preparation or re-verified unless otherwise specified in the analytical method.

Solutions may not be assigned an initial expiration interval longer than that of the component with the shortest expiration date.

6.10.4.3 Standard Reference Material Solutions

Standard reference materials should be discarded after their expiration/discard date; however, standard reference materials may be re-verified and used past their expiration/discard date for qualitative analysis only.

Stock standard and internal stock standard solutions should be deemed expired/discarded one (1) year from the date of preparation or earliest expiring standard. For qualitative analysis only, the solution may be re-verified and used past this date unless otherwise specified in the analytical method.

Dilute standard solutions (working standards including frozen calibrators) should be deemed expired/discarded one (1) year from the date of preparation. For qualitative analysis only, the solution may be re-verified and used past this date unless otherwise specified in the analytical method.

6.10.5 Blank Blood

Drug free synthetic or blank blood is obtained to match similar matrix case samples.

Drug-free whole blood can be obtained from the Oklahoma Blood Institute or purchased from a company that provides controlled blank blood.

Analyze each lot of blank blood using a full ELISA panel, basic drug screen, and any other specific analysis appropriate for its use such as an acid and neutral screen.

Store per manufacturer's recommendation.

6.10.6 Synthetic Urine

Synthetic urine is obtained to match similar matrix case samples for some analyses.

Drug-free synthetic urine can be obtained/purchased from Immunalysis or a company that provides synthetic urine.

If a COA is obtained, additional function testing is not required.

Store per manufacturer's recommendation.

7. PROCESS REQUIREMENTS

The FTU is committed to providing the best quality service available to the stakeholder. Key components to providing this level of service is implementation of a documented proficiency testing program and the use of CRMs, RMs, validated testing methods, quality controls, technical and administrative reviews, and other quality assurance practices.

7.1 Technical Procedures and Methods

The FTU will use appropriate technical procedures and methods that have been scientifically validated and/or accepted for use in the field of forensic science, are fit-for-purpose for the testing being performed, and meet the needs of the stakeholder(s). This includes methods and procedures for the handling, transport, storage, and preparation of testing items, the operation of all relevant equipment, and an estimate of the measurement uncertainty where appropriate.

7.2 Method Validation

All new methods shall be validated in accordance with OSBI CSD Quality Manual 7.2 and QP 21.2 – Evaluation of Methods, Instruments, Equipment, and Software.

The parameters to be evaluated for validation of methods will depend upon the circumstances in which the method is to be used. Likewise, it is recognized that after validation has occurred, methods may be revised. The extent and frequency of revalidation of previously validated methods will depend upon the nature of the intended changes.

The guidelines below will be used to introduce new methods or significantly modify existing methods. Contemporary scientific subcommittee guidelines should also be considered in this process.

Throughout the following procedures, the term CRM is used. If a CRM is not available, a reference material (standard) may be used instead, for qualitative purposes only. Prior to use, the RM should be analyzed by GC/MS and the spectrum checked against an outside spectral source such as a journal or validated library. If the RM is confirmed for use, then the RM may be used for qualitative validation purposes.

7.2.1 Procedure

The proper validation of a new method requires assessing the selectivity and limitations of the method as well as predicting possible sources of error. The validation process should address the baseline characteristics of precision, accuracy, selectivity, and sensitivity of the method. Records should be kept throughout the validation process. Documentation should include parameters that were evaluated during method development, even if acceptable results were not obtained.

- A validation plan shall be in place prior to starting any validation studies. The validation plan shall be followed and documented as per OSBI CSD Quality Manual QP 21.2 – Evaluation of Methods, Instruments, Equipment, and Software. The plan shall include the instrumental method(s) and sample preparation technique(s) to be used for a specific method. Further, it shall document the validation requirements of the method, as well as the limits of the method that will allow it to be fit for use. The validation plan should also include identification of individuals working on the validation. This must be approved by the FTU TM per CSD QM QP 6.2.6.
- Validation studies can be conducted by the scientific community (as in the case of standard or published methods) or by the FTU (as in the case of laboratory-developed methods, standard methods used outside their intended scope, or where significant modifications are made to previously validated methods).

Validation will be sufficient to ensure the reliability of the method against any documented performance limitations.

- The method must be tested using known samples. If a new method is intended to supersede an existing one, or if it parallels an existing one, then the two may be compared on split samples, where practical.
- If the analysis provides quantitative data, the validation should include investigation of the range, accuracy, and precision of the method relative to its intended use and the needs of the stakeholder. Uncertainty of measurement will also be evaluated, when applicable.
- Validation experiment parameters are based on the scope of the method.
    - Recommendation for Screening (Immunoassay-based) Methods:
        - Limit of detection
        - Precision (at the decision point)
        - Processed sample stability (if applicable)
    - Recommendation for Screening (all other methods targeting specific analytes or analyte classes) Methods:
        - Interference studies
        - Limit of detection
        - Ionization suppression/enhancement [for applicable techniques, such as liquid chromatography/mass spectrometry (LC/MS)]
        - Processed sample stability (if applicable)
    - Recommendation for Qualitative Confirmation Methods:
        - Carryover
        - Interference studies
        - Ionization suppression/enhancement (for applicable techniques, such as LC/MS)
        - Limit of detection
        - Processed sample stability (if applicable)
    - Recommendation for Quantitative Methods:
        - Bias
        - Calibration model
        - Carryover
        - Interference studies

- Ionization suppression/enhancement (for applicable techniques, such as LC/MS)
- Limit of detection
- Limit of quantitation
- Precision
- Dilution integrity (if applicable)
- Processed sample stability (if applicable)

Table 2 Experiments for Validation of Methods

Bias and Precision[1] (Section 7.2.3)							
• 3 concentration levels (low, medium, high), triplicate analyses (separate samples) of each concentration in 5 separate runs with new calibration curve for each run.							
Run	Calibration	Low	Mid	High	LOD	LOD	Dilution integrity Bias & Precision
1	6	3	3	3	3	3	3
2	6	3	3	3	3	3	3
3	6	3	3	3	3	3	3
4	6	3	3	3	-	-	3
5	6	3	3	3	-	-	3
Calibration Model[1] (Section 7.2.4)							
• 6 concentration levels. 5 replicates of each (may be accomplished in the same or separate calibration curves or generated for studies mentioned below).							
Carryover (Section 7.2.5)							
• 1 fortified sample with high concentration of analyte with no internal standard (IS) is alternately analyzed with blank matrix samples in triplicate (i.e., fortified sample, blank, fortified sample, blank, fortified sample, blank).							
Interference (Section 7.2.6)							
• 10 different blank sources of each matrix, no internal standard (IS) • 1 blank sample with IS • 1 fortified sample with high analyte concentrations without IS							

- Neat, fortified, or authentic samples containing potentially interfering compounds/metabolites but not the analyte(s) of interest

Ionization Suppression/Enhancement[2] (Section 7.2.7)

Post-Column Infusion

- Solutions at low and high concentrations are infused post-column.
- 10 blank extracts injected during infusion of the solutions.

Post-Extraction Addition

- Two neat standards prepared at a low and high concentration. Each is injected a minimum of 6 times to establish a mean peak area for each concentration.
- 10 blank extracts fortified after extraction at the low concentration. 10 blank extracts fortified after extraction at high concentration. Each injected once.

Limit of Detection (LOD) (Section 7.2.8)

Immunoassays

- Decision point – mathematically estimate the LOD concentration of and drugs with a cross-reactivity of less than 100% that are declared as detectable to stakeholders.

Lowest Non-Zero Calibrator (Quantitative Methods)

- Three (3) different blank matrix sources (per matrix type) fortified with the analyte at the concentration of the lowest calibrator and analyzed over a minimum of three (3) runs.

Decision Point Concentration (Quantitative and Qualitative Methods)

- Three (3) different blank matrix sources (per matrix type) fortified at the concentration of the decision point and analyzed over a minimum of three (3) runs.

Estimated Using Background Noise

- Reference Materials – Three (3) different blank matrix sources (per matrix type) fortified at decreasing concentrations shall be analyzed in duplicate (two separate samples) for at least three (3) runs.
- Statistical Analysis of Background – Three (3) different blank matrix sources (per matrix type) analyzed in duplicate (two separate samples) over at least three (3) runs. Three (3) different blank matrix sources (per matrix type) are fortified with decreasing concentration and analyzed in duplicate (two separate samples).
- Linear Calibration Curve (quantitative methods using a linear calibration model only) – minimum of three (3) independent calibration curves constructed across

the working range over different runs. A single matrix source (per matrix type) may be used.
Limit of Quantitation (LOQ)[1] (Section 7.2.9)
Lowest Non-Zero Calibrator • Three (3) different blank matrix sources (per matrix type) fortified with the analyte at the concentration of the lowest calibrator analyzed over three (3) runs. Can use same calibrator replicates used to establish calibration model. Decision Point Concentration • Three (3) different blank matrix sources (per matrix type) fortified with the analyte at the concentration of the decision point analyzed over three (3) runs.

[1] Quantitative methods only

[2] LC/MS-MS methods only

7.2.2 Conducting Method Validation Studies

All validation experiments shall be conducted using fortified samples of the matrix for which the method is intended, unless otherwise noted. In some instances, (e.g., immunoassay screens), it may be more appropriate to analyze previously characterized human samples instead of fortified samples for selected method validation studies.

When feasible, validation studies may be conducted in a manner similar to casework. This may include conducting validation studies on different days, by different analysts, and ensuring that instruments meet the same daily performance requirements as for casework.

For validation studies which identify compounds that have a similar chemical structure, molecular weight, and mass spectra, the protocol established should require use of optimized multiple reaction monitoring (MRM) transition parameters, chromatographic conditions, and may also apply product ion scanning which is automatically triggered once an MRM exceeds a specified threshold to ensure accurate identification of compounds.

When feasible, fortified matrix samples should be prepared from reference materials that are from a different source (e.g., supplier or lot number) than used to prepare calibration samples.

The following requirements are the minimum for assessing the listed validation parameters in forensic toxicology methods. They are listed alphabetically and not necessarily in procedural order.

7.2.3 Bias and Precision

Bias

Bias studies shall be carried out for all quantitative methods. These can be conducted concurrently with precision studies.

Bias shall be measured in pooled fortified matrix samples using a minimum of three (3) separate samples per concentration at three (3) different concentration pools (low, medium, high) over five (5) different runs. Low concentrations should be no more than three (3) times the lowest end of the working range of the method and high concentrations should be within approximately 80% (or more) of the highest end of the working range of the method, unless otherwise noted. Medium concentrations should be near the midpoint of the low and high concentrations. The bias shall be calculated for each concentration using the following formula:

$$Bias\ \%\ at\ Conc_x = \left[\frac{Grand\ Mean\ of\ Calculated\ Conc_x - \ Nominal\ Conc_x}{Nominal\ Conc_x}\right] \times 100$$

NOTE: In some instances, analyte instability may preclude the ability to use concentration pools of fortified samples. These instances may require fortifying different samples with each run.

The maximum acceptable bias is ± 20% at each concentration. For some analyses, where less bias is required (e.g., ethanol), a bias of ± 10% or better is expected. It is recommended that the same data used in bias studies also be used for precision calculations.

Precision

Precision studies shall be carried out for all quantitative methods, as well as the decision point for immunoassays. These studies can be carried out concurrently with bias studies, if required in the validation plan.

Precision is expressed as the coefficient of variation (% CV). The mean standard deviation(s) of the response is calculated for each concentration to determine the % CV.

$$\%\ CV = \left[\frac{standard\ deviation}{mean\ response}\right] \times 100$$

Precision of Immunoassays at Decision Point

At a minimum, precision at the immunoassay's decision point (i.e., cutoff concentration) should be assessed using three (3) separate samples per concentration at three (3) different concentration pools over five (5) different runs:

- generally, no more than 50% below decision point,

- at decision point, and
- generally, no more than 100% above decision point.

The intent of this requirement is to ensure that there is evidence that the assay separates presumptive positive and negative samples at the decision point.

Calculate the grad mean (n=15) and related grand standard deviation for each concentration pool.

The % CV shall not exceed 20% at each concentration using all 15 sample results per concentration.

The grand mean plus or minus two standard deviations of the low and high concentration pools should not overlap with the mean of the decision point.

Precision of Quantitative Procedures

For quantitative procedures, two (2) different types of precision studies shall be assessed during method validation: within-run precision and between-run precision. At a minimum, precision shall be assessed using three (3) different samples per concentration at three (3) different concentration pools (low, medium, and high) over five (5) different runs. The different runs used to evaluate precision may be performed within the same day, provided a different calibration curve is used for each run.

The % CV shall not exceed 20% at each concentration. It is noted that certain analytical methods (e.g., blood alcohol analysis) may require a much lower coefficient of variation (≤ 10% CV).

Within-Run Precision Calculations

Within-run precision is calculated for each concentration separately for each of the five (5) runs. Within-run precision may be calculated using the data from each run's triplicate analyses at each concentration as:

$$Within - Run\ CV(\%) = \frac{std\ dev\ of\ a\ single\ run\ of\ samples}{mean\ calculation\ value\ of\ a\ single\ run\ of\ samples}\ x\ 100$$

The largest calculated within-run % CV for each concentration will be used to assess within-run precision acceptability.

Between-Run Precision Calculations

Between-run precision is calculated for each concentration over the five (5) runs. This may be done by using the combined data from all replicates of each concentration as:

$$Between - Run\ CV(\%) = \frac{std\ dev\ of\ grand\ mean\ for\ each\ concentration}{grand\ mean\ for\ each\ concentration}\ x\ 100$$

The largest calculated between-run % CV for each concentration will be used to assess between-run precision acceptability.

7.2.4 Calibration Model

The calibration model shall be determined for all quantitative methods. This is accomplished by first determining the working range. Within this range, there will be a correlation between signal response (e.g., peak area ratio of analyte and internal standard) and analyte concentration in the sample. The calibration model is the mathematical model that describes correlation. The choice of an appropriate model (i.e., linear or quadratic) is necessary for accurate and reliable quantitative results.

Calibrator samples are analyzed to establish the calibration model. The use of matrix-matched calibrator samples is encouraged, but not required (i.e., water vs urine). A single source of blank matrix (per matrix type) may be used when experimentally establishing the appropriate calibration model for a method. Regardless of the matrix used to prepare calibrator samples, the laboratory shall demonstrate acceptable bias and precision with control samples prepared in all matrices intended to be analyzed by the method. For example, blood alcohol methods may demonstrate acceptable bias and precision in whole blood controls using aqueous calibrator samples. Likewise, acceptable bias and precision may be demonstrated using calibrator samples prepared in whole blood, but used to quantitate analytes in different matrices (e.g., serum, urine).

The calibrator samples shall span the range of concentrations expected in day-to-day operations. At least six (6) different, non-zero concentrations shall be used to establish the calibration model, with the exception of ethyl alcohol analysis which shall have at least five (5) different, non-zero concentrations. The concentrations shall be appropriately spaced across the calibration range to establish the most appropriate calibration model. A minimum of five (5) replicates per concentration shall be used. The replicates to establish the calibration model may be in the same or separate runs. All data points from the five (5) replicates shall be plotted together (using a statistical software package) to establish the calibration model. The origin shall not be included as a calibration point.

The simple linear regression model using the least squares method is the most often used calibration model. However, this model is only applicable when there is constant variance over the entire concentration range. When there is a notable difference between variances at the lowest and highest concentrations, a weighted least squares model or other appropriate non-linear model should be applied. This is generally the case when the concentration range exceeds one order of magnitude. Ultimately, the

simplest calibration model that best fits the concentration-response relationship should be used.

Although it has become a widespread practice, it is emphasized that a calibration model shall not be evaluated simply via its correlation coefficient (r). Therefore, a calibration model may be visually evaluated using standardized residual plots. These allow one to check for outliers that must be eliminated if found to be statistically significant (e.g., outside ± 3 standard deviations). Further, residual plots allow one to determine if the variances appear to be equal across the calibration range with a similar degree of scatter at each concentration. They also give an indication if the chosen model adequately fits the data. For example, random distribution of individual residuals around the zero line (homoscedasticity) suggest that a linear model is appropriate.

If a linear calibration model has been established, fewer calibration samples (i.e., fewer levels) may be used for routine analysis. The calibration data shall include the lowest and highest calibration levels used to establish the model, as well as include no fewer than four (4) non-zero calibration points.

Additionally, once the calibration model is established for a validated method, it shall not be arbitrarily changed to achieve acceptable results during a given analytical run.

7.2.5 Carryover

Analyte carryover into a subsequent sample may lead to an inaccurate qualitative or quantitative result when using instrumental methods. Carryover shall be evaluated during method validation intended for confirmation and/or quantitation.

To evaluate carryover as part of a method validation, blank matrix samples are analyzed immediately after a high concentration sample or reference material. The highest fortified concentration at which no analyte carryover is observed (above the method's LOD) in the blank matrix sample is determined to be the concentration at which the method is free from carryover. This carryover concentration shall be confirmed for carryover using triplicate analyses (quantitation of the concentration is not required, but calculation of the expected fortified concentration is required).

It is acceptable to limit the carryover study to the highest point of your calibration curve, but even higher concentrations are preferred. If possible, the analytical procedure should be modified to remove any carryover. In cases when it is not possible to eliminate the carryover, the standard operating procedure or the quality manual shall address how carryover will be managed.

7.2.6 Interference Studies

Interfering substances from common sources must be evaluated in all screening (except immunoassays), qualitative identification, and quantitative methods.

Evaluating Matrix Interferences

Whenever possible, blank matrix samples from a minimum of ten (10) different sources without the addition of an internal standard (when used in the method) shall be analyzed to demonstrate the absence of common interferences from the matrix. While this approach may detect the more common matrix interferences, it is recognized that less common interferences may not be detected.

Evaluating Interferences from Stable-Isotope Internal Standards

For methods employing stable isotope internal standards, the isotopically-labeled compounds may contain the non-labeled compound as an impurity. Additionally, the mass spectra of the labeled analogues may contain fragment ions with the same mass-to-charge ratios as the significant ions of the target analyte. In both instances, analyte identification or quantitation could be impacted.

Stable-isotope internal standard interferences shall be assessed by analyzing a single blank matrix sample (per matrix type) fortified with the internal standard and monitoring the signal of the analyte(s) of interest. Interferences below the LOD of the assay may be insignificant depending on the purpose of the method.

A single blank matrix sample (per matrix type) fortified with the analyte(s) at a concentration near the upper limit of the calibration range shall be analyzed without internal standard to evaluate whether relevant amounts of the unlabeled analyte ions appear as isotopically-labeled compound fragments that could impact quantitation.

Evaluating Interference from Other Commonly Encountered Analytes

For all methods other than immunoassays, it is necessary to evaluate other analytes which may be expected to be present in case samples for their potential to interfere with the method's analytes. For example, a method developed to analyze blood for cocaine shall evaluate whether other common drugs of abuse, metabolites, and structurally-similar compounds interfere with the assay. Likewise, a headspace gas chromatograph-flame ionization detector (GC-FID) method developed for ethanol must evaluate whether other common volatile organic compounds interfere with the assay.

This evaluation shall be accomplished by analyzing fortified matrix samples, previously analyzed case samples, or neat reference materials of the potential interference(s) at high therapeutic or lethal concentrations, depending on the analyte and the matrix. The

most common drugs/metabolites encountered in the laboratory must be included in the evaluation together with other common drugs within the classification, where appropriate.

7.2.7 Ionization Suppression/Enhancement

The enhancement or suppression of analyte ionization resulting from the presence of co-eluting compounds is a phenomenon commonly encountered in LC/MS applications.

When average suppression or enhancement of the analyte's target ion (or ion transition and qualifying ions) exceeds ± 25% or the % CV of the suppression/enhancement exceeds ± 20%, the laboratory shall demonstrate that there is no impact on other critical validation parameters. For example, suppression/enhancement of ionization is most likely to impact the limit of detection of a qualitative method. Likewise, the limit of detection and limit of quantitation may be affected by ionization suppression/enhancement in quantitative methods. The influence on the above parameters shall be assessed by at least tripling the number of different sources of blank matrices used in their evaluation. For example, if the average suppression/enhancement exceeds ± 25%, the LOD determination shall be performed in at least nine unique sources of blank matrices.

The impact of ionization suppression/enhancement on the method's internal standards shall also be assessed.

Ionization suppression/enhancement shall be evaluated using either of the following approaches.

<u>Post-Column Infusion to Assess Ionization Suppression/Enhancement</u>

This approach provides information on retention times where ionization suppression/enhancement occurs. It is useful for method development, as well as to assess the amount of ionization suppression/enhancement for LC/MS based confirmation methods. Solutions at both low and high concentrations of the analyte are individually infused with a syringe pump into the eluent from the column via a post-column "T" connection and a constant baseline signal for the analyte of interest is monitored. Whenever possible, a minimum of ten (10) different processed blank matrix samples (per matrix type) that are representative of the quality of samples typically encountered in casework are injected onto the LC/MS during infusion of the solutions. If there is any considerable suppression/enhancement (>25%) of the infused analyte signal at the retention time of the analyte, then modification of the chromatographic system or the sample preparation may be required to minimize the effect of ionization suppression/enhancement.

Post-Extraction Addition Approach to Assess Ionization Suppression/Enhancement

This approach yields a quantitative estimation of ionization suppression/enhancement. It is useful for assessing the amount of ionization suppression/enhancement for LC/MS based methods. Two different sets of samples are prepared, and the analyte peak areas of neat standards are compared to matrix samples fortified with neat standards after extraction or processing.

Set one consists of neat standards prepared at two concentrations – one low and one high. Each low and high concentration is injected six times. A mean peak area is calculated for each concentration.

Set two should consist of a minimum of ten (10) different matrix sources (per matrix type), whenever possible. Each matrix source is extracted in duplicate. After the extraction is complete, ten (10) of the matrix samples will be reconstituted/fortified with the low working standard and ten (10) will be reconstituted with the high working standard from "set one".

The preparation of the low and high concentrations begins by using an aliquot of a CRM diluted with a solvent to create a stock solution. The stock solution is used to prepare the working low and high concentration in an organic solvent. The organic solvent is evaporated and reconstituted with appropriate amount of reconstitution solvent.

The average area of each set ($\bar{X}$) is used to estimate the suppression/enhancement effect at each concentration as follows:

$$Ionization\ suppression\ or\ enhancement\ (\%) = \left(\frac{\bar{X}\ Area\ of\ Set\ 2}{\bar{X}\ Area\ of\ Set\ 1} - 1\right) * 100$$

Two ionization suppression/enhancement percentages will be established – one at the low concentration and one at the high concentration.

7.2.8 Limit of Detection

Limit of detection (LOD) studies should be carried out for all methods. There are a number of different approaches for determining the LOD. Select the approach that provides the most reasonable estimation of the LOD given the analytical instrumentation (or lack thereof) utilized in the method.

A method's LOD incorporates instrumental performance, as well as the sample matrix and inherent procedural limitations. Therefore, the LOD shall be assessed over multiple runs using fortified matrix samples from at least three different sources of blank matrix, unless otherwise indicated below.

The LOD must be determined by one of the following approaches:

Estimating LOD for Immunoassays

The laboratory may assign the decision point (i.e., cutoff concentration) as the LOD for immunoassays. Most of these assays are known to cross-react with numerous drugs (e.g., benzodiazepines, opiates) and metabolites. When a laboratory declares to their stakeholders that they are able to detect specific analytes demonstrating low cross-reactivity (less than or equal to the target analyte) using the immunoassay, they shall verify their ability to reliably detect these compounds.

As an example, a benzodiazepine immunoassay targeted for oxazepam typically has low cross-reactivities to many other benzodiazepines. If a laboratory uses the decision point (i.e., cut-off concentration) determined by the manufacturer, the laboratory shall mathematically estimate the LOD concentration for any benzodiazepine that cross-react at less than 100% and that they declare to their stakeholders they are able to detect with the immunoassay.

A single source of blank matrix (per matrix type) may be used to estimate LOD of immunoassays.

Using the Lowest Non-Zero Calibrator as the LOD

This technique is useful for quantitative methods. In some instances, it may be sufficient to define the LOD as the value of the lowest non-zero calibrator. For each matrix type, at least three different blank matrix sources shall be fortified with the analyte at the concentration of the lowest calibrator and analyzed over a minimum of three (3) runs to demonstrate that all detection and identification criteria are met. If desired, it is acceptable to use the same calibrator replicates used to establish the calibration model for some of the samples used for this approach, but additional samples/replicates may be needed to meet the minimum of nine (9) points including at least three sources per matrix type.

Using the Decision Point Concentration as the LOD

This technique is useful for qualitative and quantitative methods. In some instances, it may be sufficient to define the LOD as the value of an administratively-defined decision point. For example, a laboratory may choose to define a method's LOD for ethanol as 0.020 g/100mL for blood based on the laboratory's administratively defined decision point for reporting this analyte, even though a lower LOD is analytically achievable. For each matrix type, a minimum of three (3) different blank matrix sources shall be fortified with the analyte at the concentration of the decision point shall be analyzed over a minimum of three (3) runs to demonstrate that all detection and identification criteria are met.

Estimating LOD Using Background Noise

These approaches for determining LOD are only useful for instrumental methods that demonstrate background noise. A minimum of three (3) different blank source per matrix shall be used.

Estimating LOD Using Reference Materials

Three (3) (or more) sources of blank matrix samples (per matrix type) fortified at decreasing concentrations shall be analyzed in duplicate (two separate samples) for at least three (3) runs. The LOD is considered the lowest concentration that 1) yields a reproducible instrument response greater than or equal to 3.3 times the noise level of the background signal in an area around the analyte peak and 2) achieves acceptable predefined detection and identification criteria (e.g., retention time, peak shape, mass spectral ion ratios) for all replicates at that concentration.

While it may be possible to visually assess the signal–to–noise ratio, such an approach is subjective. Therefore, calculate the signal–to-noise ratio or use instrumentation software to determine the ratio. If manually calculated, the signal is defined as the height response of the analyte peak and the noise is defined as the amplitude between the highest and lowest point of the baseline in an area around the analyte peak. Each replicate shall be independently evaluated.

$$Signal - to - Noise = \frac{height\ of\ analyte}{amplitude\ of\ noise}$$

Estimating LOD Using Statistical Analysis of Background

To determine the LOD using this approach, a minimum of three (3) sources of blank matrix samples shall be analyzed in duplicate (two separate samples) over at least three (3) runs. The average and standard deviation(s) of the signal (e.g., integrated area of signal at the analyte's retention time) from all negative samples is calculated. Likewise, fortified samples of decreasing concentration are analyzed in duplicate over the course of at least three (3) runs. The lowest concentration of a fortified sample that consistently yields a signal greater than the average signal from the blank matrix samples plus 3.3 times the standard deviation of the signal from the blank matrix samples, shall be identified as the LOD.

$$LOD = x + 3.3s$$

Estimating LOD Using a Linear Calibration Curve

This technique is useful for any quantitative method that follows a linear calibration model. A minimum of three (3) independent calibration curves shall be constructed

across the working range of the analytical method over different runs. A single source of blank matrix (per matrix type) may be used to estimate the LOD using this approach. The LOD can be estimated from the standard deviation of the *y* intercept (s_y) and the average slope (Avg_m) as:

$$LOD = (3.3\ s_y)/Avg_m$$

7.2.9 Limit of Quantitation

Limit of quantitation (LOQ) studies shall be carried out for all quantitative methods. There are a number of different approaches for determining a method's LOQ. Select the approach that provides the most reasonable estimation of the quantitation limit given the analytical instrumentation utilized in the method. A method's LOQ incorporates instrumental performance, as well as the sample matrix and inherent procedural limitations. The LOQ shall be assessed over multiple runs using fortified, blank matrix samples from at least three (3) different sources per matrix type, unless otherwise indicated below.

Using the Lowest Non-Zero Calibrator as the LOQ

In some instances, it may be sufficient to define the LOQ as the value of the lowest non-zero calibrator. For each matrix type, minimum of three (3) different blank matrix sources shall be fortified with the analyte at the concentration of the lowest calibrator and analyzed over a minimum of three (3) runs to demonstrate that all detection, identification, bias, and precision criteria are met. If desired, it is acceptable to use the same calibrator replicates used to establish the calibration model for this approach, but additional samples/replicates may be needed to meet the minimum of nine (9) data points including three sources per matrix type.

Using Decision Point Concentration as the LOQ

In some instances, it may be sufficient to define the LOQ as the value of an administratively defined decision point. For example, the laboratory may choose to define a method's LOQ for GHB as 5 mg/L for antemortem blood based on the laboratory's administratively defined decision point for reporting this analyte, even though a lower LOQ is analytically achievable. The concentrations used for this approach shall remain within the previously established calibration curve. For each matrix type, a minimum of three (3) different blank matrix sources shall be fortified with the analyte at the concentration of the decision point shall be analyzed over three (3) runs to demonstrated that all detection, identification, bias, and precision criteria are met.

7.2.10 Additional Validation Parameters

In certain instances, it is important to evaluate additional validation parameters, if applicable. These include: processed sample stability of the analyte(s) and the effect of sample dilution on bias and precision. The laboratory shall include these parameters in the laboratory's validation plan and determine if they are applicable to the analytical method or if they are already addressed through other means (i.e., quality assurance practices).

7.2.11 Dilution Integrity

The effect of sample dilution shall be determined during validation of quantitative methods if this is a routine practice within the laboratory. At times, this may be due to low specimen volume requiring the sample or assay to be adjusted appropriately. In other instances, excessively high concentrations that are above the established calibration range may be encountered. To bring the analyte concentration within the validated concentration range, the laboratory procedure may allow for reanalysis after dilution of the sample.

If dilution of a sample is allowed because of high analyte concentration or low sample volume, then the laboratory must evaluate the effect of dilution on the method's bias and precision with at least one concentration pool. This shall be accomplished by establishing bias and within-run precision studies at common dilution ratios (e.g., 1:2, 1:10, 1:50) utilized by the laboratory and determining if performance criteria are still met.

7.2.12 Stability

Analyte stability may be affected by a number of variables, including storage conditions and sample processing. Stability experiments shall be designed and carried out to address situations normally encountered in laboratory operations, unless analyte stability is already addressed through other means (i.e., quality assurance practices, published references). All stability determinations shall include a set of samples prepared from reference materials. The reference materials are used to prepare fortified samples of the analyte(s) at both low and high concentration in each matrix that will be analyzed in the method. It is important that a large enough volume of each of these fortified samples is prepared in order to complete the studies used in the sections below. These fortified samples shall initially be analyzed in triplicate to establish zero responses. The average time zero response for each set of samples is compared to the average signals from each of the following stability studies. Linear regression of the average signal (e.g., peak areas or ratios of peak area of analyte to internal standard) versus time will allow for an assessment of trends.

Stability – Processed Sample

Circumstances may arise in which samples that have undergone routine preparation for instrumental analysis cannot be immediately analyzed. It may be necessary to run the sample the following day or later. In these instances, it is important to evaluate the length of time a processed sample can be maintained before it undergoes unacceptable changes, preventing reliable analyte detection, identification, or quantitation.

Typically, processed fortified samples are combined per concentration and then divided into different autosampler vials. As indicated above, the first vials of each concentration are immediately analyzed in triplicate to establish the time zero responses. All remaining vials are stored in a manner that they would typically be stored during routine analysis (e.g., refrigerated, at room temperature on autosampler). The remaining vials are then analyzed in triplicate at different time intervals. Average responses at each time interval are compared to the time zero responses. The analyte will be considered stable until the average signal (e.g., peak area or ratios of peak area of analyte to internal standard) compared to the time zero average signal falls outside of the method's acceptable bias. For example, a method's bias limit is ± 15% and the time zero average signal is 100,000. Processed samples in different autosampler vials are analyzed repeatedly up to 72 hours. The processed sample's analyte is considered stable until the average signal falls outside of the 85,000 – 115,000 range.

7.2.13 Required Revalidation of Previously Validated Methods

Modifications to a validated method require evaluation to confirm that the changes do not have an adverse effect on the method's performance. The decision regarding which performance characteristics require additional validation is based on logical consideration of the specific parameters likely to be affected by the change(s). These changes may include, but are not limited to:

- Analytical conditions
- Instrumentation
- Sample processing
- Data software

For example, changes of extraction solvent or buffer may affect linearity, interferences, LOQ, precision, and bias. A change of the analytical column stationary phase or a change in the mobile phase composition may affect linearity and interferences. Further, consideration should be given to conducting parallel studies with known or proficient samples utilizing both a previously validated method and the modified method in order

to evaluate the effects of the changes. The goal is to demonstrate the impact the changes have on the performance of the previously validated procedure.

7.2.14 Documentation Requirements for Method Validation

Record keeping is a key component of method validation. The data generated during method validation studies must be maintained and available for audits, reviews, or inspections.

Method validation records must include a summary of the validation studies conducted and their results. The format of the summary report may be brief bulleted report or table summary format to facilitate a swift review of validation studies. The summary shall minimally include the following: scope, validation plan, and description of all the parameters evaluated.

If any of the parameters were not evaluated, then the reason should be stated or justified.

Other information that should be included (if applicable):

- Sample preparation steps to include concentrations and matrices
- Raw data or reference to where the raw data is stored
- Results and calculations
- Conclusions
- References
- Documentation of management review and approval

It is important that the validation record contain specific details regarding the studies conducted, including:

- Individuals involved in the method validation
- Specific instrumentation
- Dates

Method validation documentation shall also include a copy of the newly developed analytical method or a reference to its location.

7.2.15 Efficiency With Validation

It is recognized that method validation is a time-consuming, expensive, but essential endeavor. Keep in mind that some validation experiments may be conducted concurrently with the same fortified samples.

7.3 Sampling

Vials included in blood kits are generally collected at the same time and are, therefore, considered to be the same sample (one blood draw deposited into separate vials).

For cases that contain different types of samples (e.g., blood and urine) or different vial types (e.g., hospital samples), the items should be sub-itemized and only the items analyzed will have reported results. All untested items should be reported as not analyzed.

Ensure that all samples are homogenous by shaking, rotary mixing, and/or vortexing. A tissue grinder can be used to break up any clots.

If a homogenous sample cannot be obtained for any reason, a notation shall be made in the LIMS detailing the condition of the sample and its handling.

Factors that shall be controlled in analysis of toxicology evidence include: specimen pretreatment, selection of aliquot container, proper labeling of aliquot container, selection of pipette/pipette dilutor, volume to be sampled, and handling of aliquots.

7.4 Sample Handling and Storage

7.4.1 Receiving Samples

Physical Evidence Quality Procedures Manual PE QP 1 – Evidence Intake, outlines evidence intake procedures. Toxicology DUI/D evidence is typically submitted by mail through a carrier service (United States Postal Service, United Parcel Service, or Federal Express). Evidence may also be received in person from submitting officers. Specimens should be received and in-processed by the Physical Evidence Technicians and/or Criminalists trained to log-in evidence. Once received, the Physical Evidence Technicians may deliver evidence to the FTU as well as the Officer's Affidavits/RFLEs and any other accompanying documentation.

Physical Evidence Quality Procedures Manual PE QP 2.1 – Evidence Handling, outlines evidence handling procedures used by OSBI CSD employees during the examination of evidence. If a significant discrepancy between the evidence received and the description provided is discovered, consult Physical Evidence Quality Procedures Manual PE QP 2.1 – Evidence Handling, prior to analysis.

7.4.2 DUI/DUID Evidence

Typically, three (3) vials of whole blood are submitted for cases requiring toxicological analysis. If less than three vials of sample are submitted, the criminalist shall identify and prioritize analyses in order to maximize the value of the toxicological analyses.

Specimens of blood collected under the provisions of Title 47, Title 3, and Title 63, of the Oklahoma Statutes should be collected in 10 milliliter (mL) gray top, glass vacuum vials labeled by the manufacturer as containing 100 milligrams (mg) of sodium fluoride and 20 mg of potassium oxalate. Such containers are approved by the Board of Tests and supplied by the OSBI for the collection of blood for analysis for the presence or concentration of ethyl alcohol, the presence of intoxicating substances, or a combination of the two.

It is recognized that hospital or clinical specimens collected pursuant to medical treatment may be collected in blood vials with or without preservatives. An appropriate description of the evidence will be included on the report. The description of the evidence may also be noted in the LIMS or on a respective worklist and included in the case record.

The following are descriptions, per the 2010 BD Vacutainer Venous Blood Collection Tube Guide, of some of the vials that may be encountered:

Cap Color		Additive	Inversions Recommendation	Laboratory Use
 Gold	 Red/ Gray	• Clot activator and gel for serum separation	5	For serum determinations in chemistry. May be used for routine blood donor screening and diagnostic testing of serum for infectious disease.** Tube inversions ensure mixing of clot activator with blood. Blood clotting time: 30 minutes.
 Light Green	 Green/ Gray	• Lithium heparin and gel for plasma separation	8	For plasma determinations in chemistry. Tube inversions ensure mixing of anticoagulant (heparin) with blood to prevent clotting.
 Red	 Red	• Silicone coated (glass) • Clot activator, Silicone coated (plastic)	0 5	For serum determinations in chemistry. May be used for routine blood donor screening and diagnostic testing of serum for infectious disease.** Tube inversions ensure mixing of clot activator with blood. Blood clotting time: 60 minutes.
 Orange		• Thrombin-based clot activator with gel for serum separation	5 to 6	For stat serum determinations in chemistry. Tube inversions ensure mixing of clot activator with blood. Blood clotting time: 5 minutes.
 Orange		• Thrombin-based clot activator	8	For stat serum determinations in chemistry. Tube inversions ensure mixing of clot activator with blood. Blood clotting time: 5 minutes.
 Royal Blue		• Clot activator (plastic serum) • K_2EDTA (plastic)	8 8	For trace-element, toxicology, and nutritional-chemistry determinations. Special stopper formulation provides low levels of trace elements (see package insert). Tube inversions ensure mixing of either clot activator or anticoagulant (EDTA) with blood.
 Green	 Green	• Sodium heparin • Lithium heparin	8 8	For plasma determinations in chemistry. Tube inversions ensure mixing of anticoagulant (heparin) with blood to prevent clotting.
 Gray	 Gray	• Potassium oxalate/ sodium fluoride • Sodium fluoride/Na_2 EDTA • Sodium fluoride (serum tube)	8 8 8	For glucose determinations. Oxalate and EDTA anticoagulants will give plasma samples. Sodium fluoride is the antiglycolytic agent. Tube inversions ensure proper mixing of additive with blood.
 Tan		• K_2EDTA (plastic)	8	For lead determinations. This tube is certified to contain less than .01 µg/mL(ppm) lead. Tube inversions prevent clotting.
	 Yellow	• Sodium polyanethol sulfonate (SPS) • Acid citrate dextrose additives (ACD): **Solution A -** 22.0 g/L trisodium citrate, 8.0 g/L citric acid, 24.5 g/L dextrose **Solution B -** 13.2 g/L trisodium citrate, 4.8 g/L citric acid, 14.7 g/L dextrose	8 8 8	SPS for blood culture specimen collections in microbiology. ACD for use in blood bank studies, HLA phenotyping, and DNA and paternity testing. Tube inversions ensure mixing of anticoagulant with blood to prevent clotting.
 Lavender	 Lavender	• Liquid K_3EDTA (glass) • Spray-coated K_2EDTA (plastic)	8 8	K_2EDTA and K_3EDTA for whole blood hematology determinations. K_2EDTA may be used for routine immunohematology testing, and blood donor screening.*** Tube inversions ensure mixing of anticoagulant (EDTA) with blood to prevent clotting.
 White		• K_2EDTA and gel for plasma separation	8	For use in molecular diagnostic test methods (such as, but not limited to, polymerase chain reaction [PCR] and/or branched DNA [bDNA] amplification techniques.) Tube inversions ensure mixing of anticoagulant (EDTA) with blood to prevent clotting.
 Pink	 Pink	• Spray-coated K_2EDTA (plastic)	8	For whole blood hematology determinations. May be used for routine immunohematology testing and blood donor screening.*** Designed with special cross-match label for patient information required by the AABB. Tube inversions prevent clotting.
 Light Blue  Clear	 Light Blue	• Buffered sodium citrate 0.105 M (=3.2%) glass 0.109 M (3.2%) plastic • Citrate, theophylline, adenosine, dipyridamole (CTAD)	3-4 3-4	For coagulation determinations. CTAD for selected platelet function assays and routine coagulation determination. Tube inversions ensure mixing of anticoagulant (citrate) to prevent clotting.
 Clear	 New Red/ Light Gray	• None (plastic)	0	For use as a discard tube or secondary specimen tube.

Substitutions to types of blood vials used for collection in the provided kit will be included on the report with an appropriate description of the evidence. Sub-itemization may be necessary to provide this information. The substitution may also be noted in the LIMS or on a respective worklist and included in the case record.

For example:

- Three 4 mL gray top vials were substituted for the provided 10 mL gray stopper vials can be written:
    - "Item 1: One sealed OSBI Blood Specimen Collection Kit, blood kit number ###### containing three unsealed 4 mL gray top vials containing whole blood."
    - Sub-itemization is not necessary.
- An evidence envelope contains one red stopper vial and 2 gray stopper vials can be written:
- "Item 1: One sealed evidence envelope labeled "John Doe" containing items 1A-1B.
  Item 1A: Two unsealed gray stopper vials containing blood.
    - Item 1B: One unsealed red stopper vial containing blood. Sub-itemization would be necessary.

In general, the specimens with the earliest collection times should be analyzed whenever possible.

7.4.3 Drug-Facilitated Sexual Assault Evidence

Blood should be collected in addition to urine, preferably within 48 hours of the alleged incident.

Typically, two vials of whole blood are submitted for cases requiring toxicological analyses. If less than two vials of blood are submitted, the criminalist shall identify and prioritize the analyses in order to maximize the value of the toxicological analyses.

Blood samples, when feasible, should be collected in gray top vials (or equivalent) containing 100 mg of sodium fluoride and 20 mg of potassium oxalate to prevent degradation and clotting.

Urine should be collected in any case in which the complainant reports within the first 120 hours (5 days) after the alleged assault. A minimum of 50 mL of urine should be collected in a sterile container. If less than 50 mL of urine are submitted, the criminalist will identify and prioritize the analyses in order to maximize the value of the toxicological analyses.

It is recognized that hospital or clinical specimens collected pursuant to medical treatment may be collected in blood vials with or without preservatives. An appropriate description of the evidence will be included on the report. The description of the evidence may also be noted in the LIMS or on a respective worklist and included in the case record.

Substitutions to types of blood vials used for collection in the provided kit will be included on the report with an appropriate description of the evidence. The substitution may also be noted in the LIMS or on a respective worklist and included in the case record.

In general, the specimens with the earliest collection times should be analyzed whenever possible.

7.4.4 Other Drug Related Crime (i.e., poison, child endangerment, etc.)

At least two, 5 mL blood samples, when feasible, should be collected in gray top vials (or equivalent) containing 100 mg of sodium fluoride and 20 mg of potassium oxalate to prevent degradation and clotting.

Urine samples should be collected in a standard sterile urine collection cup.

It is recognized that hospital or clinical specimens collected pursuant to medical treatment may be collected in blood vials with or without preservatives. An appropriate description of the evidence will be included on the report. The description of the evidence may also be noted in the LIMS or on a respective worklist and included in the case record.

Substitutions to types of blood vials used for collection in the provided kit will be included on the report with an appropriate description of the evidence. The substitution may also be noted in the LIMS or on a respective worklist and included in the case record.

In general, the specimens with the earliest collection times should be analyzed whenever possible.

7.4.5 Labeling

For the toxicology Blood Specimen Collection Kits, the following labeling technique should be used when feasible:

Label blood vials with the OSBI laboratory case number, the item number (e.g., 1) and an identifier beginning with the letter “A” to unambiguously differentiate the vials from one another, the date the kit is opened, and the analysts initials. The vial containing the largest volume of blood should be labeled “A”, when

feasible. The vial containing the second largest volume of blood should be retained as the defendant's sample and labeled "C", when feasible. The final vial should be labeled "B". The estimated volumes for each vial should be recorded in the LIMS or on a respective worklist and included in the case record.

For the toxicology Drug Facilitated Specimen Collection Kits, the following labeling technique should be used when feasible:

Label blood vials with the OSBI laboratory case number, the item number (e.g., 1A) and an identifier beginning with the number "1" to unambiguously differentiate the vials from one another (e.g., 1A1, 1A2), the date the kit is opened, and the analyst's initials. The vial containing the largest volume of blood should be labeled "1", when feasible. The second vial of blood should be labeled "2", when feasible. Label the urine collection cup with the OSBI laboratory case number, the item number (e.g., 1B), the date the kit is opened and the analyst's initials. The estimated volumes for each vial should be recorded in the LIMS or on a respective worklist and included in the case record.

If a technician inventories a kit, both the technician and the criminalist shall initial each blood vial. The evidence shall also be transferred to the technician's custody while being inventoried. After the evidence has been inventoried, it shall remain in the custody of the criminalist until the report has been issued.

For all non-kit cases requiring sub-itemization, consult Physical Evidence Quality Procedures Manual PE QP 2.1 – Evidence Handling.

7.4.6 Case Assignments

Cases awaiting analysis without an assignment are not reflected on the open case listing which does not accurately reflect the current workload in the FTU. Any casework in the FTU awaiting any type of analysis should have an open assignment to reflect the status. If an additional request has been made by phone, email, or in person, for an additional analysis, it is the responsibility of the analyst taking the message to create the assignment and document the conversation at that time in the case narrative. If additional analysis will not be performed, it should be documented in a case narrative and conveyed to the caller.

7.4.7 Handling Test Item(s)

A worklist is to be prepared whenever samples are to be tested. The worklist will indicate the case numbers and should be used to compare the worklist to the case numbers during preparation of samples for analysis in order to ensure that all samples

have been properly selected. Worklists containing any notes will be attached to the batch run in the LIMS.

When analyzing samples, indication of which item/vial was examined shall be made. Notation can be made on attached data or in the LIMS. Since blood alcohol analysis and presumptive drug screens typically utilize the vial labeled “A” or “1” (e.g., 1A or 1A1), no note is required for these tests, unless a vial other than “A” or “1” is used.

7.4.8 Storage

Biological specimens received by physical evidence units and the FTU should, as appropriate, be refrigerated (1 – 10°C) as soon as possible to preserve their condition.

Whenever evidence is not actively being analyzed, it should be stored in evidence refrigerators.

7.4.9 Retention and Storage of Blood Specimens

Each State’s blood specimen, in its sealed container and employing other shipping or transport enclosures as required, should be promptly dispatched or forwarded by the law enforcement agency to the FTU of the OSBI.

Whenever a State’s blood specimen is collected under the provisions of O.S. Title 47, at the direction of a law enforcement officer and for the purpose of determining the concentration of alcohol or presence of other intoxicating substance(s) thereof, an additional and separate blood specimen should be collected. The resulting additional specimen is termed “Retained Blood Specimen” or “Retention Specimen”. The retention specimen has been labeled “C” and will not be used for analysis by the FTU, when feasible.

The tested person or their attorney may direct retention specimens for independent analysis, within 60 days from the date of collection, to be forwarded to a laboratory of their own choosing and approved by the Board of Tests for an independent analysis in accordance with O.S. §47-752. Neither the tested person, nor any agent of such person, shall have access to the retained blood specimen prior to the completion of the independent analysis, except the analyst performing the independent analysis and agents of the analyst.

Each Retention Specimen, shall be kept and stored by the OSBI FTU for at least 60 days from the date of collection (O.S. §47-752), unless transferred prior thereto to an independent laboratory. The independent analysis of blood will be at the expense of the tested person whose option it is to have such analysis performed. The tested person, or their attorney, shall make all necessary arrangements for the performance of such

independent analysis other than the forwarding or delivering of such specimen (O.S. §47-752).

After the expiration of the 60 days from the date of collection, all such retention specimens, other than those transferred to an independent laboratory, may be promptly and safely destroyed (40:20-1-3 Oklahoma Administrative Code) by the OSBI laboratory.

Upon completion of the report, all blood kits should be sealed and should be kept in long term storage for a minimum of four months after the date received. Specimens can be released to the requesting agency if requested by that agency. Hospital or clinical specimens collected pursuant to medical treatment for the purpose of O.S. 47 §751, may be disposed of in the same manner.

7.4.10 Documenting Limited Samples

Per OSBI CSD Quality Manual QP 16.2 – Contents of Case Records, the Prosecutor in the case will be advised by the examining criminalist prior to limited quantity samples being analyzed and consumed. A letter from the appropriate prosecuting attorney authorizing the consumption of those samples will be placed in the case record.

The evidence will be photographed. A ruler or size standard will be included in the photograph, if feasible.

Photographic documentation will be included in the case record.

Every reasonable attempt will be made to comply with any special request regarding the analysis of limited quantity evidence. These contacts and any special requests should be documented in an appropriate fashion (memo, e-mail, narrative, etc.) in the case record. The Division Director should be notified of any special requests.

7.5 Technical Records

7.5.1 General Recordkeeping

The case record examination documentation must be sufficient to support the reported results, conclusions, interpretations, and/or opinions per OSBI CSD Quality Manual QP 16.2 – Contents of Case Records.

Any administrative or technical documentation which is required to be retained, but is not readily incorporated into the LIMS case record shall be scanned into the case record. Results of alcoholic beverage content cases may be documented in the LIMS system.

7.5.2 Start Dates & End Dates

The FTU will consider the “start date” of analysis as the date the sample inventorying process begins, while the “end date” of analysis will be the date the reviewing criminalist approves the report in the LIMS.

7.5.3 Sample Tracking

The security and integrity of all evidence in the possession of the FTU will be preserved. Transfer, return, and/or destruction of evidence will be conducted according to the specifications of Physical Evidence Quality Procedures Manual PE QP 3 – Evidence Transactions.

7.5.4 Rejected Data

Data not used for interpretation, such as an initial scan that was rejected (e.g., controls not acceptable, etc.) will be retained on the instrument it was analyzed on as stated in OSBI FTU Quality Manual 4.2. Likewise, a file may be created for the case and called “rejected data” and a pdf of the data placed there.

If an observation, data, or calculation is rejected, the reason, the date, and the identity of the individual taking the action shall be recorded in the criminalist’s notes. For example, “MNB 8/14/25: case sample 25-01234 data on instrument F was rejected due to poor chromatography”.

7.5.5 Tracking Changes to Technical Records

Amendments to technical records shall follow OSBI CSD Quality Manual 7.5.2.

Mistakes/changes in examination documents should be crossed out with a single line, initialed, and corrected.

File names may be corrected on the instrument, but a copy of the original raw data will also be saved without the correction.

All case records should accurately reflect the OSBI laboratory case number and item/vial used for analysis. Any typos discovered after analysis are not required to be corrected on the instrument. See OSBI FTU Quality Manual section 7.4.7 for additional information.

Examination documentation is considered complete when it has been submitted for administrative/technical review.

Any changes made to completed examination documentation shall be tracked.

7.6 Uncertainty of Measurement

Uncertainty of measurement will be applied to a specification limit (stated by case law, statute, or legal requirement), external to the FTU. Measurement uncertainty for all quantitations will be included on the report.

The purpose of this section is to provide technical direction for the FTU to meet ISO/IEC 17025 requirements. This will serve as the FTU's document describing the estimation of uncertainty when it impacts evaluation of a specification limit external to the FTU.

Calibration certificates with valid uncertainties must be available for all calibrated equipment, reference standards, and reference materials. These are supplied by the supplier.

Statistical data regarding the calibration measurement process must be available; preferably from a measurement control program (i.e., historical data) and available from the FTU's in-house measurement control process.

Knowledge of the technical basis for the measurement is critical for completeness in the evaluation of measurement uncertainty. This can be obtained through reference papers, reference procedures, brainstorming, experimentation, inter-laboratory comparisons, cause and effect diagrams, and the like.

Each measurement made has a corresponding uncertainty assigned to the measured value. The uncertainty is directly related to the measurement parameter (scope), range of the measurement, the equipment or measurement process being used (affecting precision), and the standards available with associated uncertainties.

7.6.1 Uncertainty Analysis Process

This uncertainty analysis process used the following eight steps:

1) Specify the measurement process;
2) Identify and characterize uncertainty components;
3) Quantify uncertainty components in applicable measurement units;
4) Convert uncertainty components to standard uncertainties in unit of the measurement result;
5) Calculate the combined uncertainty;
6) Expand the combined uncertainty using an appropriate coverage factor;
7) Evaluate the expanded uncertainty against appropriate tolerances, user requirements, and laboratory capacities; and
8) Report correctly rounded uncertainties with associated measurement results.

7.6.2 Specify the Process

Define the measurand by inputting the following information into a budget form in Excel labeled “Measurement Uncertainty Estimation Form” found in the “Toxicology_Lab” folder on the server.

1) Measurement (e.g., Concentration of ethanol in whole blood)
2) Range of measurement values (e.g., 0.010 g/100mL to 0.400 g/100mL)
3) Procedure name and revision (e.g., Procedure No. TX05, Revision 11)
4) Estimation prepared by (e.g., John Doe)

7.6.3 Identify and Characterize Uncertainty Sources

1) Identify and compile a list of the possible uncertainty components that may have an influence on the measurement process (e.g., components that affect analytical measurement uncertainty may include: analysts, calibrators, QC samples, sampling, preparation, and testing). Store the list in the “Toxicology_Lab” folder on the server.
2) Identify all possible sources of uncertainty in a comprehensive list, characterizing them based on the evaluation method that will be used to quantify them (Type A, statistical methods or Type B, scientific judgement) and transfer the sources of uncertainty to a spreadsheet and store in the “Toxicology_Lab” folder on the server.
3) The uncertainty components can be grouped into two categories. Characterize the components based on the evaluation method (Type A, statistical methods or Type B, non-statistical methods). Enter “A” or “B” for the type of method into the column labeled “Type” on the budget form.

7.6.4 Quantify Uncertainty Estimates

1) Estimate the relative uncertainty and express it as a percentage associated with each component and enter that numerical value into the column labeled “Value” on the budget form.
2) ‘Type A’ evaluations are based on statistical methods requiring the calculation of the mean, $\bar{x}$, and standard deviation, s. From these, the percent relative standard deviation, %RSD, is calculated (refer to Calculations section).

   ‘Type B’ evaluations are based on non-statistical information using all the relevant information available which is derived from such sources as calibration certificates or tolerance limits (refer to Calculations section).

   For example, a calibration certificate uncertainty for a CRM is 0.000233 g/100mL for the 0.010 g/100mL CRM use %RSD as follows:

   % Relative uncertainty = (0.000233 g/100mL/0.010 g/100mL) * 100 = 2.33%

Therefore, 2.33 would be input into the column labeled “Value” on the budget form.

3) The values for percent relative uncertainty must be expressed in the form of a percentage therefore place a “%” in the column labeled “Units” on the budget form.

4) Not all of the documented uncertainty components will have a significant contribution to the overall uncertainty. In the budget form, a line or strike through may be placed through any insignificant contributors. Be sure to provide an explanation in the “The basis for data above” section that it was considered, yet determined to be insignificant.

5) To consider an uncertainty component significant, it should cause a change in the value of the second most significant digit, leading zeros excluded, when included in the uncertainty calculations. For example, if the expanded uncertainty value is currently 0.052 g and including the value of an uncertainty contributor causes the new value to be 0.053 g, that contributor is considered significant. If no change in the second significant digit results, the ‘2’, the contributor is not considered significant.

6) Identify the probability distribution for ‘Type A’ and ‘Type B’ uncertainty components and enter the type into the column labeled “Distribution” on the budget form. In general, most real-world measurement applications are normal, rectangular, or Student’s t distributions. Some recommendations for selecting the appropriate distribution are as follows:

    a. Normal distribution, ‘Type A’, should be applied when a collection of repeat measurements of a quantity of interest are presented such as historical positive control data.

    b. Normal distribution, ‘Type B’, should be applied when calibration certificates contain the “expanded uncertainties” and is divided by the coverage factor (k) to obtain the standard uncertainty.

Table 3. Typical Coverage Factors

Coverage Factor (k)	Level of Confidence (%)	Divisor
1	68.27	1
1.960	95	1.960
2	95.45	2
3	99.73	3

    c. Rectangular distribution should be applied if limits of ± are given without a confidence level (e.g., A 10 mL, Grade A, volumetric flask is certified to within ± 0.2 mL. the standard uncertainty is $0.2/\sqrt{3}$ = 0.11 mL).

    d. Student’s t distribution typically arises when the measurement process lacks a sufficient number of measurements (n) i.e., historical data. A sample size of

30 at 29 degrees of freedom or higher, the t-distribution begins to approximate the normal (Gaussian) distribution. Apply the student's t distribution when measurements are n<30. Thus, for an analysis lacking historical data, a corrected coverage factor is used based on the student's t table (NIST Technical Note 1297).

For example, for an analysis with low historical control data, a control is analyzed 15 times (degrees of freedom or df = n-1, or 14 for this example).

Using the student's t table, k_{corr} value of 2.20 would be used to calculate the expanded uncertainty at 2σ or 95.45% confidence limit.

e. If the probability distribution cannot be determined, then treat it as a rectangular distribution.

7.6.5 Distribution and Divisor

1) The "Divisor" is determined from the probability distribution that was entered into the column labeled "Distribution" and documented in the column labeled "Divisor".

Table 4. Distribution and Divisors

Distribution	Divisor
Type A – Normal	Use table 5 and degrees of freedom
Type B – Normal	See Table 3 and coverage factor from calibration certificate
Type B – Rectangular	$\sqrt{3}$
Student t when n<30	Use table 5 and degrees of freedom

2) Degree of freedom is used as an indication of the reliability of the uncertainty value. When the student's t table is used then enter the numerical value for degrees of freedom into the column labeled "Degrees Freedom (n-1)" on the budget form.

3) Once the "value", "Distribution", and "Divisor" for each source of uncertainty has been determined, then the standard uncertainty for all sources can be calculated.

   Convert all factors to standard uncertainties.

4) Convert the standard uncertainties by dividing the relative uncertainty inputted into the "Value" column by its respective "Divisor" and enter that numerical value into the column labeled "Standard Uncertainty" on the budget form.

5) The "Measurement Uncertainty Estimation Form" will calculate this numerical value.

7.6.6 Calculate the Combined Uncertainty

After calculating the standard uncertainties, the combined standard uncertainty can be calculated. Determine the combined standard uncertainty, $u_c(y)$, simply by calculating

the square root of the sum of each standard uncertainty squared (RSS). The "Measurement Uncertainty Estimation Form" will calculate this numerical value.

$$u_c(y) = \sqrt{u_1^2 + u_2^2 + u_3^2 + u_4^2} \ldots\ldots$$

7.6.7 Calculate the Expanded Uncertainty

1) The combined standard uncertainty is then multiplied by a coverage factor, k, based on the degrees of freedom, to provide a level of confidence of 95.45% or 99.73%, depending on what is required by the stakeholder (the FTU currently uses 99.73%). The equation used to determine the expanded uncertainty is as follows:

$$U = u_c * k$$

2) The coverage factor must be determined from a statistical table such as provided in the Guide to the Expression of Uncertainty in Measurement or NIST Technical Note 1297.

3) After the expanded uncertainty, U, has been determined, the numerical value will be rounded up to two significant digits.

   Example:

   k = 2.000(95.45%) U = 4.3074 * 2.000 = 8.6148% = 8.7%

   k = 3.000(99.73%) U = 4.3074 * 3.000 = 12.9222% = 13%

   If the degrees of freedom are 100 or below, then use the student's t table for the respective k value. The k value determined at 95.45% and 99.73% are multiplied into the combined standard uncertainty to obtain the "reported uncertainty".

7.6.8 Evaluate the Expanded Uncertainty

The expanded uncertainty may be evaluated against established criteria such as tolerance limits, stakeholder requirements, and/or calibration and measurement capabilities on the laboratory scope. For example, the expanded uncertainty must not exceed 20% of the prior year's calculation. The budget should be reviewed annually by the FTU Technical Manager or designee.

7.6.9 Report the Uncertainty

When reporting measurement uncertainty, the value shall be reported in the Criminalistics Examination Report, shall be expressed as an expanded uncertainty, and include the coverage probability of 99.73%.

This measurement result shall include the measured quantity value, y, along with the associated expanded uncertainty, U. This measurement result shall be reported as y ± U using the same units of measurement.

Example:

BAC = 0.090 g/100mL

Using k = 3 (U = 10%); 0.10 * 0.090 g/100mL = 0.009 g/100mL

The measurement result and the rounded expanded uncertainty shall be reported in the same units and to the same level of significance.

Example report statement:

Ethyl Alcohol Content: 0.090 ± 0.009 g/100mL

Additionally, the coverage probability will be found in the Report Summary.

7.6.10 Expanded Uncertainty Significant Digits and Rounding

Numerical values of expanded uncertainties shall be no more than two significant figures. The following applies:

- The numerical value of the measurement result shall be rounded to the least significant figure in the value of the expanded uncertainty, U.
- When rounding, examine the digit following (i.e., to the right of) the digit that is to be the last digit in the rounded off number. The digit you are examining is the first digit to be dropped.
    - If the digit immediately to the right of the last significant figure is LESS than 5, the last significant figure is unchanged.
    - If the digit immediately to the right of the last significant figure is EQUAL TO OR GREATER than 5, the last significant figure is rounded up.

7.6.11 Calculations

Historical control data is a 'Type A' component requiring these calculations:

- For n measurements of the historical data x, the **mean** value is:

$$\overline{x} = \frac{1}{n}\sum_{i} x_i$$

- The standard deviation is found by averaging the squares of the deviations, and then taking the square root:

$$s = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \overline{x})^2}{n-1}}$$

- The mathematical expression for **relative standard deviation** (RSD):

$$RSD = \frac{s}{|\overline{x}|}$$

- The percent relative standard deviation:

$$\%\ RSD = \text{RSD}*100$$

- The mathematical expression for **relative standard deviation** of the mean:

$$RSD_{mean} = \frac{s}{\sqrt{n}}$$

- The percent relative standard deviation of the mean:

$$\%\ RSD_{mean} = RSD_{mean}*100$$

When 'Type B' components have been identified and the uncertainty is given, it should be used. Typically, uncertainties given on calibration reports will be "expanded uncertainties" usually using a "coverage factor of k=2". An "expanded uncertainty" is the standard deviation (or "standard uncertainty") which has been multiplied by a number called the "coverage factor". For example, a calibration certificate for a mass standard states:

> "The uncertainty in the reported mass is ± 26 mg at a level of confidence of 95% assuming a normal distribution." The standard uncertainty is ± 26 mg ÷ 1.960 = ± 13 mg (1.960 from NIST Technical Note 1297); or
>
> "The expanded uncertainty in the reported mass with a coverage factor, k, of 2." is ± 26 mg ÷ 2 = ±13 mg.

7.6.12 Technical Note 1297

Value of tp(*v*) from the t-distribution for degrees of freedom, *v*, that defines an interval tp(*v*) to + tp(*v*) that encompasses the fraction, p, of the distribution.

NOTE: table is taken from NIST Technical Note 1297.

Table 5. Student's t table

Degrees of freedom ν	Fraction *p* in percent					
	68.27(a)	90	95	95.45(a)	99	99.73(a)
1	1.84	6.31	12.71	13.97	63.66	235.80
2	1.32	2.92	4.30	4.53	9.92	19.21
3	1.20	2.35	3.18	3.31	5.84	9.22
4	1.14	2.13	2.78	2.87	4.60	6.62
5	1.11	2.02	2.57	2.65	4.03	5.51
6	1.09	1.94	2.45	2.52	3.71	4.90
7	1.08	1.89	2.36	2.43	3.50	4.53
8	1.07	1.86	2.31	2.37	3.36	4.28
9	1.06	1.83	2.26	2.32	3.25	4.09
10	1.05	1.81	2.23	2.28	3.17	3.96
11	1.05	1.80	2.20	2.25	3.11	3.85
12	1.04	1.78	2.18	2.23	3.05	3.76
13	1.04	1.77	2.16	2.21	3.01	3.69
14	1.04	1.76	2.14	2.20	2.98	3.64
15	1.03	1.75	2.13	2.18	2.95	3.59
16	1.03	1.75	2.12	2.17	2.92	3.54
17	1.03	1.74	2.11	2.16	2.90	3.51
18	1.03	1.73	2.10	2.15	2.88	3.48
19	1.03	1.73	2.09	2.14	2.86	3.45
20	1.03	1.72	2.09	2.13	2.85	3.42
25	1.02	1.71	2.06	2.11	2.79	3.33
30	1.02	1.70	2.04	2.09	2.75	3.27
35	1.01	1.70	2.03	2.07	2.72	3.23
40	1.01	1.68	2.02	2.06	2.70	3.20
45	1.01	1.68	2.01	2.06	2.69	3.18
50	1.01	1.68	2.01	2.05	2.68	3.16
100	1.005	1.660	1.984	2.025	2.626	3.077
α	1.000	1.645	1.960	2.000	2.576	3.000
For a quantity *z* described by a normal distribution with expectation μz and standard deviation σ, the interval $\mu z \pm k\,\sigma$ encompasses *p* = 68.27, 95.45, and 99.73 percent of the distribution for *k* = 1, 2, and 3, respectively.						

7.7 Ensuring the Validity of Results

7.7.1 Reinjection Criteria and Documentation

Occasionally, there may be situations in which calibrators, controls, and case samples need to be reinjected on a chromatographic instrument. Common reasons for reinjection include the following:

- Poor analyte or internal standard recovery.
- Sample overload.
- Poor chromatography or interference.

When a sample is reinjected, document that the original injection was unacceptable in the case record, using language such as “not used due to (reason for reinjection)”. Include initials and date. The rejected raw data will be stored on the instrument as stated in FTU QM 4.2.

7.7.2 Carryover Determination

- Carryover is evaluated by comparing the ratio of the abundance of the analyte in the sample to the negative control.
- For GC/MS full scan – If carryover is noticed in a negative control or case sample and meets identification criteria listed in FTU QM – Drug Identification Criteria, the criminalist should review possible reasons for carryover (e.g., saturated detector in previous sample, wash vials low, liner change needed). If a reason for the carryover is apparent, it should be corrected and the sample reinjected. If a reason is not apparent, maintenance may be needed (see FTU QM – Gas Chromatograph Mass Spectrometers). The sample should be reinjected. If carryover is still present, the criminalist will either request that the FTU Technical Manager, or designee, review the data for acceptability or reanalyze the sample(s).
- For GC/MS Selected Ion Monitoring (SIM) and LC/MS Multiple Reaction Monitoring (MRM) – The peak area in the case sample must be at least 10 times greater than the peak area in the negative control. The peak area in the low positive control must also be at least 10 times greater than the peak area in the negative control. If the peak area in the low positive control or the case sample is less than 10 times the peak area in the negative control, the data for that analyte will not be used for reporting purposes.

7.7.3 Control Charts (QC logs)

Quality control materials are assayed and the quality control results are inspected to assure the quality of the analytical run. This documentation is accomplished for ELISA

(Tecan) and quantitations by maintaining a quality control (QC) log and using the Levy-Jennings chart on a regular basis. The QC logs can be found in the "Toxicology_Lab" folder on the server. Once QC results are entered into the QC log, they should be plotted on the Levy-Jennings chart automatically if the chart is in use.

For ELISA (Tecan), results of the positive control (%B/B0) will be entered into the ELISA QC log in Excel which will plot the value. The values should be documented after analysis and will be documented before administrative and technical reviews.

For alcohol analysis, results of the positive control (0.080 g/100mL), low blood control, and high blood control values will be entered into the alcohol analysis QC log in Excel which will plot the value for the positive control. The values should be documented after the calibration run and will be documented before administrative and technical reviews.

When the results are plotted, an assessment can be made about the quality of the run. The criminalist performing the test should look for systematic error and random error.

Systematic error is evidenced by a change in the mean of the control values. The change in the mean may be gradual and demonstrated as a trend in control values or it may be abrupt and demonstrated as a shift in control values.

A trend indicates a gradual loss of reliability in the test system. Trends are usually subtle.

Abrupt changes in the control mean are defined as shifts. Shifts in QC data represents a sudden and dramatic positive or negative change in test system performance.

Random error is any deviation away from an expected result. For QC results, any positive or negative deviation away from the calculated mean is defined as random error. There is acceptable (or expected) random error as defined and quantified by standard deviation. There is unacceptable (unexpected) random error that is any data point outside the expected population of data (e.g., a data point outside the ± 3 standard deviation limits).

7.7.4 Drug Identification Criteria

The identification of drugs in biological specimens is normally a two-step process: initial testing (or screening) followed by confirmation testing. Initial testing is normally accomplished by chromatographic methods, immunoassay methods, or chemical color tests, either singly or in some combination depending on the scope of the screening protocol. Confirmation testing is normally accomplished using a mass spectrometric method. Confirmation testing is performed, when feasible, on an aliquot of the specimen separate from that used for screening.

Analytical results used for identification should come from at least two different methods whenever feasible. For drug confirmation, one of the methods shall be a mass spectrometric method. Should two different methods not be feasible, confirmation testing on another aliquot of the same specimen by repeat analysis shall be done. When multiple concurrent specimens are available from the same person (e.g., blood and urine, hospital vials with the same collection time regardless of cap color), duplicate testing of each specimen is not required. For example, the following practices are acceptable:

- Blood alcohols by GC analysis based on duplicates.
- Identification of cocaine in blood or urine with a single GC/MS or LC/MS-MS analysis in combination with immunoassay screening of a separate aliquot of the same specimen.
- Identification of diphenhydramine in blood or urine based on duplicate GC/MS or LC/MS-MS analyses.
- Confirmation of trazodone in blood by a single GC/MS or LC/MS-MS analysis when a paired urine specimen has been determined positive for trazodone by GC/MS or LC/MS-MS analysis.
- Confirmation of dextromethorphan in two different hospital vials, same or different colored tops, collected at the same time, by GC/MS or LC/MS-MS analysis.

7.7.4.1 Headspace GC/FID Identification Criteria

Drug Identification Criteria

The retention times of ethyl alcohol and internal standard peaks in the sample must be within ± 2% of the positive control sample.

The results of duplicate case specimens must be within ± 5% of the average of the duplicate results or the case specimen must be reanalyzed.

Should one result be above the high calibrator and one result be below, the results must be within 5% of each other. For example, if the results are 0.402 and 0.387, calculate 5% of the lower result (0.019) and verify that the higher result is within range (0.387 + 0.019 = 0.406).

Control Acceptance Criteria

The multicomponent volatile mix solution shall have separation of the components including defined, symmetrical peaks for each of the five components and the internal standard. Only components of the multicomponent volatile mix solution and the internal standard shall be present.

The calibration standards must be within 0.002 g/100mL of the 0.010 g/100mL control and ± 5% of all other expected calibration control concentrations and should be free of all analytes of interest except ethyl alcohol. The calibration curve for ethanol shall have a correlation of determination r2 ≥ 0.995.

The negative control shall be free of all analytes of interest.

The positive control shall quantitate within ± 5% of the target value (0.080 g/100mL) for ethyl alcohol and should be free of all analytes of interest except ethyl alcohol.

The low and high whole blood controls will quantitate within ± 20% of the expected concentration provided by the manufacturer.

All verification solutions will be within ± 5% of the expected concentration (0.150 g/100mL).

If the criteria for the calibrators or controls are not met, then the calibration shall be repeated using freshly prepared controls and calibrators with acceptable results prior to ethanol being identified, confirmed, or quantitated.

If a verification solution used to bracket cases is outside the acceptable tolerance, all cases bracketed by that verification solution will be reanalyzed and bracketed with verification solutions that meet the above acceptance criteria.

Table 6. Calibrator and Control Target Concentrations

Target Concentration (g/100mL)	Acceptable Tolerance Range (g/100mL)
0.010	0.008 – 0.012
0.100	0.095 – 0.105
0.200	0.190 – 0.210
0.300	0.285 – 0.315
0.400	0.380 – 0.420
0.500	0.475 – 0.525
0.080	0.076 – 0.084
0.150	0.142 – 0.158

7.7.4.2 ELISA Identification Criteria (Tecan)

Drug Identification Criteria

Positive ELISA results should be reviewed and samples scheduled for appropriate confirmatory testing.

Control Acceptance Criteria

If there is no separation between the positive control (PC) and high positive control (HPC) on the pdf included in the case record, or the HPC is a higher number than the

PC (e.g., the PC is 25 and the HPC is 30) then the data for that test should be reanalyzed. The negative control (NC) should be 100.

NOTE: If a result more negative than the negative control is observed (e.g., a result of 200 or more), the results should be viewed with more scrutiny since this could indicate an issue with the analysis.

A run is evaluated when a single control measurement exceeds the mean ± 3 standard deviation of control limits. Deviations from the rejection of data may occur based on the individual circumstance. When a single data control value is outside ± 3 standard deviations, the criminalist should decide to either:

- reanalyze the samples; or
- request that the FTU Technical Manager, or designee, review the data control value for acceptability.
    - If the FTU Technical Manager, or designee, reviews the data, they will evaluate the control value to determine if no relationship can be found and no source of error can be identified.
    - If no relationship is identified, then the single data value may be viewed as an accepted random error.

Factors that should be evaluated include:

- distance from the mean,
- separation of the low and high controls,
- the frequency of positive samples identified in the analytical run,
- if a lot change has occurred, and
- the type of error.

If the samples are reanalyzed and a deviation continues to occur outside ± 3 standard deviations two consecutive times, the FTU Technical Manager, or designee, should be notified.

The FTU Technical Manager, or designee, shall evaluate the process and document the evaluation and any action taken in the instrument maintenance log or QC log.

7.7.4.3 Headspace GC/MS Identification Criteria

Drug Identification Criteria

The retention times of the compound and internal standard peaks in the sample must be within ± 2% of the compound and internal standard peak(s) in the positive control that is analyzed the same day.

The mass spectrum of the target compound in the case sample should compare favorably to that of the mass spectrum of the compound in the positive control that is analyzed the same day.

The mass spectrum should contain all the major and diagnostic ions unique to that analyte. Each analyte should also form a symmetrical peak.

Control Acceptance Criteria

The positive control should contain the internal standard and the drug(s) contained in the working positive control only. “Junk” peaks (e.g., nitrogen, phthalates, methanol for CRMs/RMs made in methanol) do not impact the acceptability of the positive control. If the primary standard was in methanol, the presence of methanol in the positive control does not impact the acceptability of the control. Each analyte should also form a symmetrical peak.

The negative control should be free from any analytes being tested for, except internal standards. “Junk” peaks (e.g., nitrogen, phthalates) do not impact the acceptability of the negative control. Each analyte should also form a symmetrical peak.

If the positive and/or negative control(s) do not meet the acceptance criteria, ethyl alcohol/drugs will not be identified or confirmed.

7.7.4.4 Qualitative GC/MS Identification Criteria

Drug Identification Criteria

Each analyte should form a symmetrical peak with a height that is about five times the baseline.

The retention indices (RI) for target compounds should be ± 25 RI units of those in the OSBI spectral library. A greater tolerance may be acceptable at the discretion of the FTU Technical Manager, or designee, on a case-by-case basis.

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

NOTE: Missing ions or the presence of additional ions in the unknown sample is indicative of a weak signal, background noise, or co-eluting substances.

The retention indices are calculated by data analysis using the following equation:

$$RI = \left(\left[\frac{\text{Rt(peak of interest)} - \text{Rt(preceding n} - \text{alkane)}}{\text{Rt(following n} - \text{alkane)} - \text{Rt(preceding n} - \text{alkane)}} + C_n \right] (\text{preceding n} - \text{alkane}) \right) * 100$$

The identification of a drug shall compare favorably with the spectral library match (OSBI toxicology library) or comparison to an actual standard reference material.

"Junk" peaks (e.g., fatty acids, phthalates, hydrocarbons, etc.) are not to be reported.

The scan range for a full scan shall begin at 40 m/z to at least 550 m/z. A partial scan may begin at an m/z value greater than an abundant ion due to the derivatizing agent (e.g., the m/z 73 ion arising from trimethylsilyl derivatives).

The mass spectrum should contain all the major and diagnostic ions unique to the analyte.

Control Acceptance Criteria

The positive control should contain the internal standard(s) and the drugs contained in the working positive control only. "Junk" peaks (e.g., fatty acids, phthalates, hydrocarbons) do not impact the acceptability of the positive control. Peaks should meet the qualitative GC/MS drug identification criteria.

The negative control should be free from any analytes being tested for, except internal standards. "Junk" peaks (e.g., fatty acids, phthalates, hydrocarbons) do not impact the acceptability of the negative control.

If the positive and/or negative control(s) does not meet the acceptance criteria, drugs will not be identified or confirmed (see FTU Quality Manual – Carryover Determination).

7.7.4.5 Qualitative LC/MS Identification Criteria

Drug Identification Criteria

Each analyte should form a symmetrical peak with an analyte peak area:internal standard peak area ratio that is at least 20% of the analyte peak area:internal standard peak area ratio present in the low positive control. See table 7 for limits of detection for each compound in TX42.

Table 7. Limit of detection for each compound in TX42.

Compound	of LPC for Blood	of LPC for Urine
4-ANPP	100%	100%
4-FIBF/PFBF	100%	50%
Acetyl Fentanyl	25%	25%
Acryl Fentanyl	100%	50%
Alpha-Methyl Fentanyl	100%	100%
Butyryl Fentanyl	100%	100%

Cyclopropyl/Crotonyl Fentanyl	25%	25%
Fentanyl	25%	25%
Fluorofentanyl	100%	100%
Furanyl Fentanyl	100%	100%
Methoxy Acetyl Fentanyl	25%	25%
Norfentanyl	100%	100%
Sufentanil	50%	25%
Valeryl Fentanyl	25%	25%

The retention times for the analyte of interest(s) and the standards/controls must agree within ± 0.15 minutes of those in the low positive control. These are inclusive ranges.

The MRMs (i.e., precursor and products) being monitored are within their given retention time windows.

The calculated ion ratios will be set within 30% relative to the average of the positive controls. All samples and controls will be analyzed under the ion ratio set for that batch run.

Control Acceptance Criteria

The low and high positive controls should contain the internal standard(s) and the drugs contained in the working positive controls. Peaks should meet the qualitative LC/MS drug identification criteria.

The negative control should be free from analytes being tested for, except internal standards.

If the low or high positive and/or negative control(s) does not meet the acceptance criteria, drugs will not be identified or confirmed (see FTU Quality Manual – Carryover Determination).

7.7.5 Background Subtraction and Computer-Based Spectral Library Matching

The presence of additional ions in the mass spectrum may be indicative of background noise or a co-eluting substance. Attempt to isolate the source of the additional ions and subtract prior to searching the reference collection of reference material mass spectra. Criminalists may include a scan prior to background subtraction in the case record. Background subtracted scans should be identified as such [e.g., BSB or (-)].

Recent advances in computer-assisted peak resolution using the mass spectral data have been established. One example of such a program is the Automated Mass Spectral Deconvolution and Identification System (AMDIS) this application is provided by the

vendor, such as Agilent® and part of the software package. This program is permitted for laboratory use.

Selected Ion Monitoring Mode

In cases where the response of a suspicious substance is weak, it may be necessary to acquire selected ions in order to detect the substance.

When selected ions are monitored, at least two analyte qualifying ions and one internal standard qualifying ion are preferred. For analytes with limited qualifying ions, it is minimally acceptable to use one qualifying ion in addition to the target ion.

7.7.6 Data Interpretation – Thermally Labile Compounds

In the event a degradation product such as desoxychlordiazepoxide is observed without an associated peak from the parent compound (chlordiazepoxide), the sample must be reinjected on another instrument to confirm the presence of the parent compound prior to reporting. The specimen may also be extracted and analyzed by LC/MS-MS for the parent compound.

7.7.7 Technical/Administrative Reviews

When a criminalist reviews each case prior to routing it for technical review, they should follow the steps below as well as verify any difference between documents by either calling the requesting officer, looking up the subject in On Demand Court Records or the Oklahoma State Courts Network, or contacting National Crime Information Center (NCIC). Make changes in the LIMS if necessary and add a narrative indicating changes.

Reviews will be conducted in accordance with OSBI CSD Quality Manual QP 31 – Reviews, on all test reports and examination records prior to release to the stakeholder.

Administrative and technical documentation shall be documented in accordance with OSBI CSD Quality Manual QP 16.2 – Contents of Case Records. **Examination records shall be considered completed prior to technical review and any changes made to technical/examination records during or after the review process must be recorded.** Technical reviews shall not be conducted by the author(s) or co-author(s) of the examination records or test report under review. All technical reviews will also consist of an administrative review. Toxicology casework is 100% technically reviewed.

The technical review shall include, but is not limited to, verification of the following:

- Compare the arrestee's name, officer's name, blood kit number, and any other pertinent information from the copy of the blood kit to the affidavit and any other enclosed documents (i.e., DRE form, RFLE, etc.). Compare these documents to the report.

- Verify citation number in the LIMS and on the affidavit, if applicable. Review the chain of custody, ensuring the criminalist has or had custody of the evidence.
- Read all narratives for pertinent information.
- Ensure all documents (for correct case) are attached in the LIMS.
- Review document from instrumental analyses (i.e., GC/MS and LC/MS-MS). Ensure all pages have the criminalist initials (if required), laboratory number, and that barcodes match (if used). Ensure the notes are properly numbered and contain the phrase “pg 1 of _” or similar wording (if required).
- Mathematical calculations performed for a case must be technically reviewed per OSBI CSD Quality Manual 7.11.6. A signature on the technical review may replace the required initials.
- Review scans and make sure criteria set forth in policy are met. For example, GC/MS RIs are within 25 units, case sample and library spectra match, drug(s) reported from FTU library only.
- If two extractions were performed, compare results from each as well as the reported results to ensure all drugs were correctly reported.
- Review the “notes” button to ensure all necessary notes are included.
- Review results to ensure the ethyl alcohol, ELISA, and extraction results are recorded correctly. Review the approximate volumes, number of vials, position numbers, notes, etc. Ensure that any presumptive screen performed by ELISA that was not confirmed is noted correctly, if necessary, on the report.
- Review “extra information” button for each batch for any batch notes.
- Verify that reagents are attached for each batch.
- Review list of completed methods and verify that they match the analyses completed for the case.

Should the reviewer identify discrepancies, or determine that additional testing is needed in the process of the technical review, the case record will be routed to the criminalist. The reviewer will communicate to the criminalist and document, through the routing function in the LIMS, those necessary change(s) or the additional test(s) requested.

The reviewer will document their technical review by using the LIMS to complete the following steps:

1) Click on the “Tech Rev” button from the assignments tab
2) Complete the checklist in the window that opens
3) Click the “Tech Rev” button in the lower right corner

4) Enter your password and click “yes”
5) The reviewer should notify the issuing criminalist that the review has been complete. Notification can be accomplished with the routing function “Report Finalized and Approved” (RFA) prior to selecting the “Approve” button and selecting “yes” in the LIMS.

Class II Nonconforming Work

Monitoring Class II nonconforming work will be followed in accordance to OSBI CSD Quality Manual QP 13 – Nonconforming Work and QP 14.1 – Nonconforming Work – Class II, which provides a process to improve upon the quality of work the FTU performs and assist in identifying developing patterns which may require attention.

If a Class II nonconformance has been identified, the reporting criminalist will notify the FTU Technical Manager, or designee, before the correction. To simplify the notification and provide a time stamp the following procedure should be followed:

1) Reviewer: Route code “C” and document in comments that it is a Class II correction, what policy was not followed, and how it can be corrected.
2) Criminalist: Route code “RTM” to route to the FTU Technical Manager regarding how the Class II nonconforming work will be handled.
3) FTU Technical Manager: Route code “RQC” for approval of Class II nonconformance correction.

Example:

1) Route “C”: “Class II – Per OSBI FTU Quality Manual 7.7.5, drug X appears present and meets acceptance criteria, but was not confirmed, needs second extraction and addition to report if confirmed”
2) Route “RTM”: “second extraction will be performed for drug X and added to report if criteria for identification is met.”
3) Route “RQC”: “Approved. M. Brous”

7.8 Reporting of Results

Reports issued by the FTU summarize analytical findings, and/or provide interpretation of toxicology results. Due to the wide variety of requests and evidence received, this section is only a general guideline for report writing. It will not always be possible to write a report using only the examples provided here. It is acceptable to use other wording as long as the results of the examinations are accurately communicated and follow similar formatting as standard reports. Recommended report writing specified in individual FTU standard operating procedures will override any guidance in this section.

Analytical reports will be prepared and issued according to OSBI CSD Quality Manual QP 28 – Report Writing. Any report being issued and requiring additional work will need to indicate, “Additional report will follow when analysis is complete” or similar wording.

Each method used should be added to the bottom of the report based on the task type(s) entered in the case. “The analysis completed in this case utilized the following methods:”.

Per OSBI CSD Quality Manual 7.8, results shall be reviewed and authorized prior to release.

7.8.1 Specimen Unsuitable for Analysis

When the condition of a submitted blood specimen does not allow for proper homogenous sampling and analysis, it should be reported as “Sample condition rendered it unsuitable for analysis” or similar wording.

7.8.2 Insufficient Specimen

When an insufficient volume of blood specimen does not allow for proper sampling and analysis, it should be reported as “Insufficient sample for analysis – no analysis performed” or similar wording.

When some testing has been completed, but there is insufficient sample to complete all testing (e.g., a second confirmation analysis), it should be reported as “additional analysis not completed due to insufficient sample” or similar wording as well as the results of the completed analyses.

7.8.3 Reporting Blood Alcohol Concentrations

Report the ethyl alcohol concentration to three decimal places of the average of the replicates in g/100mL, include the expanded uncertainty, and the coverage interval. Ethyl alcohol should be truncated when necessary.

Ex. Ethyl Alcohol Content: 0.090 ± 0.009 g/100mL.

The “Report Summary” will include the following statement, “The expanded uncertainty of the concentration is expressed at the 99.73% coverage interval.”

Should one result be above the high calibrator and one result be below, the results must be within 5% of each other and the lower result will be reported. This is based on the fact that any concentration above the top of the curve has not been validated to be linear and therefore should not be used to calculate a concentration. This also allows for a more conservative result.

Ex. Results are 0.402 and 0.387
0.387 * 5% = 0.019

0.387 + 0.019 = 0.406
Ethyl Alcohol Content: 0.387 ± 0.036 g/100mL

The presence of ethyl alcohol above the upper limit of quantitation is reported as: “Ethyl alcohol greater than [upper limit of quantitation] of blood. This result exceeds our highest calibrator concentration ([upper limit of quantitation]).”

Ex. Ethyl Alcohol greater than 0.400 g/100mL of blood. This result exceeds our highest calibrator concentration (0.400 g/100mL).

When results are negative, “None Detected” will be used.

Quantitative ethyl alcohol results ≤0.009 g/100mL, but ≥ 0.001 g/100mL will be reported as “None Identified”. The following statement shall also be added to the report in the report summary section, “a result of “None Identified” indicates that reporting requirements were not met.”

The measurement uncertainty should be reported following the “Rounding” section of the OSBI FTU Quality Manual.

Cases with an uncertainty of measurement that is equal to or less than 0.0009 g/100mL will be reported as ± 0.001 g/100mL.

If reporting serum/plasma results, the following statement should be included on the report, “serum/plasma ethanol concentrations are higher than corresponding whole blood concentrations.”

7.8.4 Reporting Urine Alcohol Results

Urine ethyl alcohol quantitative results are influenced by multiple factors including hydration of the individual, time since last bathroom use, and time since alcohol ingestion. Quantitative results also do not provide any additional insight into when alcohol was consumed, how much was consumed, or what kind was consumed. Therefore, urine ethyl alcohol quantitative results will be reported in a qualitative manner.

When results are ≥ 0.010 g/100mL, “Detected” will be used.

When results are negative, “None Detected” will be used.

When results are ≤ 0.009 g/100mL and ≥ 0.001 g/100mL, “None Identified” will be used. The following statement shall also be added to the report in the report summary section, “a result of “None Identified” indicates that reporting requirements were not met.”

7.8.5 Reporting Immunoassay Results

ELISA screen results do not need to be included on the report unless confirmation testing was not completed.

If additional tests have not been performed to confirm ELISA positive results (i.e., barbiturates, fentanyl, cannabinoids, opiates, benzodiazepines, oxycodone/oxymorphone, cocaine/benzoylecgonine), then this should be communicated to the stakeholder in the report.

Ex. A presumptive drug screen indicated a positive result for Cannabinoids for Item 1B. Note: Presumptive positive drug screen results should not be used in court.

> **NOTE**: Confirmation of the opiates, benzodiazepines, oxycodone/oxymorphone, and cocaine/benzoylecgonine plates may require LC/MS-MS testing if no opiates, benzodiazepines, oxycodone/oxymorphone, cocaine/benzoylecgonine are identified by a drug alkaline screen. If this additional analysis is not completed, this should be communicated to the stakeholder in the report.

If all ELISA results are negative, and no additional tests have been performed, then this should be communicated to the stakeholder in the report.

Ex. Presumptive Drug Screen: Negative

If a method is not available for confirmation testing of a positive presumptive result, a criminalist may choose to include a note on the report to convey this to the stakeholder. For example, if a urine sample tests presumptive positive for opiates, but no opiates are identified by TX01. TX40 is not validated for urines so the note “the OSBI Forensic Toxicology Unit currently has no validated method for opiates in urine by LC/MS-MS. Presumptive positive results cannot be confirmed and further testing for opiates cannot be performed for [Item #]” or another relevant, clear, and unambiguous statement may be used.

7.8.6 Reporting GC/MS & LC/MS-MS Results

<u>Qualitative Results</u>

When results are positive, the name of the drug will be listed followed by “– Detected.”

Ex. Item 1: Morphine, Codeine and 6-Acetylmorphine – Detected

When results are negative, “No drugs detected” or another relevant, clear, and unambiguous statement may be used.

When results do not meet reporting requirements, “No drugs identified” or another relevant, clear, and unambiguous statement may be used. The following statement shall

also be added to the report in the report summary section, “a result of “No drugs identified” indicates that reporting requirements were not met.”

The FTU does not report caffeine, nicotine, cotinine, and acridine on a standard basis.

7.8.7 Reporting Alcoholic Beverage Concentrations

Sample results must be reported as percent volume per volume (% v/v).

Report the average ethanol concentration of the duplicates truncated to the tenths place (e.g., 10.3% v/v, 3.2% v/v) include the expanded uncertainty, and the coverage interval.

The measurement uncertainty should be reported following the “Rounding” section of the OSBI FTU Quality Manual.

Ex. Ethyl Alcohol Content: 6.3 ± 0.2% v/v

When results are negative, “None Detected” or another relevant, clear, and unambiguous statement may be used.

When results do not meet reporting requirements, “None Identified” or another relevant, clear, and unambiguous statement may be used. The following statement shall also be added to the report in the report summary section, “a result of “None Identified” indicates that reporting requirements were not met.”

7.8.8 Rounding

Be sure the FTU standard operating procedures being used does not address rounding. If not, when rounding:

1) If the value of the number to the right of the rounding digit is less than five, the rounding digit is left unchanged.
2) If the value of the number to the right of the rounding digit is equal to or greater than five, the rounding digit is raised by one.
3) As an exception to the above rules, when a range is created by the acceptance criteria, the range will be rounded so that both sides of the range are equal, thus preventing partial bias for one side of the range.

    For example, the acceptance criteria for an ethanol verifier (0.150 g/100mL) is ± 5%. By following the rounding rules above, the range would be 0.143 – 0.158 g/100mL thus allowing 0.007 g/100mL below and 0.008 g/100mL above the expected value. To equal out the allowance, the range would be set as 0.142 – 0.158 g/100mL.

Results should be rounded after all calculations are complete.

7.8.9 Truncating

Although there is little statistical basis for truncating experimentally determined values, certain circumstances necessitate this process such as the truncation of reported alcoholic beverage concentration.

The process of truncating is relatively straightforward. When truncating a number, the second uncertain digit is disregarded. Furthermore, the values of the digits known with certainty along with the first uncertain digit remain unchanged.

Ex. 6.96 truncates to 6.9; 0.3435 truncates to 0.343.

Results should be truncated after all calculations are complete.

7.8.10 Opinions and Interpretations

In addition to reporting findings of toxicology examinations, FTU criminalists are often asked to interpret those findings in a court of law. Such interpretations generally fall into one of the following categories:

- Pharmacokinetic and/or pharmacodynamics principles.

  Questions posed in an individual case may include whether or not a specific dose of a drug would be detected in a toxicology specimen within a particular time period after exposure.

- Effects of drugs on the average person.

  Effects of a drug on performance are often helpful to a jury when deciding if the behavior of a suspect or a victim may have been caused by a drug. A criminalist may be asked if certain symptoms are consistent with those caused by a particular drug.

- The significance of a blood concentration of a drug.

  Reported blood concentrations may be correlated to those in the published literature to provide interpretation about whether or not the concentration is consistent with reported therapeutic, toxic, or fatal levels, if available.

References used to answer these questions vary widely. Medicolegal Aspects of Alcohol, ed. J.C. Garriott is a common first source for information related to the forensic toxicology of ethyl alcohol. Baselt's Disposition of Toxic Drugs and Chemicals in Man is a good starting point for information on other drugs and poisons. Data on effects of drugs and poisons on the average person are available in pharmacological studies, case reports, and in many other sources.

Criminalists should ensure that their opinions are congruent with current scientific standards, and not be manipulated into extending their testimony beyond the scope of

their authorization to work. The criminalist shall limit their work and testimony to the types of work they are authorized to perform. The scope of testimony is as follows:

7.8.11 Interpretation of Drug/Alcohol Results

Pharmacological effects of drugs or alcohol detected will be related to published clinical and analytical research data. Additionally, possible drug interactions may be described and their possible effects explained. There is limited scientific literature on impairment by drugs other than alcohol. Therefore, results for drugs will be interpreted as to how an average individual would or could be theoretically affected by a drug or drugs.

7.8.12 Quantitative Drug Analysis Results

Quantitation of the drug(s) found in blood samples is of limited value. There is no scientific literature that relates quantity of a drug in the blood stream to impairment, except in the instance of alcohol. If the drug(s) quantified is a prescription or over-the-counter drug, testimony may be given as to the relationship of the blood levels observed to published therapeutic, toxic, or lethal levels. There are no "therapeutic" levels for many illicit drugs (i.e., phencyclidine).

To effectively establish impairment, an interpretation of impairment should include: (a) the observation of poor driving, (b) poor field sobriety test performance, and (c) the presence of a drug or metabolite consistent with the subject's symptomology. A good case for driving impairment is established when observations of an officer or a DRE correlate with the findings of the toxicologist. The OSBI FTU does not perform quantitative analysis other than for ethanol.

7.9 Retrograde Extrapolation

A retrograde or retroactive extrapolation, is a method by which a person's blood alcohol concentration (BAC) at an earlier point in time is calculated based on their BAC from a later blood test.

> "No forensically valid forward or backward extrapolation of blood alcohol concentrations is ordinarily possible in a given subject and occasion solely on the basis of time and individual analysis results." – Dubowski.

The OSBI FTU will not perform retrograde extrapolation.

8. MANAGEMENT SYSTEM REQUIREMENTS

8.1 Performance and System Audits

Internal audits of the OSBI CSD facilities and functions will be conducted in accordance with OSBI CSD Quality Manual QP 17 - Audits.

Abbreviations	Definitions
(-)	Negative
(+)	Positive
5% pH ME Si Cap	5% phenylmethyl siloxane capillary
A/N	Acidic/Neutral Drugs
AAFS	American Academy of Forensic Sciences
ABC	Alcoholic Beverage Content
Acq.	Acquired
Act.	Actual
Alc	Alcohols/Alcohol Screen
AMPH	Amphetamine
AMT	Alli M Timmons
AN NC	Acid Neutral Negative Control
AN PC	Acid Neutral Positive Control
ANAB	American National Standards Institute National Accreditation Board
ANSI	American National Standards Institute
Appr	approximately
Approx, ~	Approximately
ASB	Academy Standards Board
ASCLD	American Society Of Crime Laboratory Directors
AT	Autotune
Ave, Avg	Average
BAC	Blood Alcohol Content
BARB	Barbiturates
BAS	Beth A Snoddy (Elizabeth)
Bases	Basic Drugs
BE	Benzoylecgonine
BEAST	Barcode Evidence Analysis, Statistics and Tracking
BENZ	Benzodiazepines
BGS, BSB	Background subtraction
Bkosbi	One sealed “OKLAHOMA BLOOD SPECIMEN COLLECTION KIT FOR ALCOHOL AND/OR DRUG DETERMINATIONS” containing
Bld	Blood
Blk	Blank
BNC	Blood Negative Control
BPC	blood positive control
Bps	brown paper sack
BslpH	Baseline peak height
BUP	Buprenorphine
BZG	Benzoylecgonine
Cal	Calibrator
CAP	College of American Pathologists
Clcap	clear capped vial
CMH	Cortney M Hanna

Con	containing
conc	concentrated/concentration
Cont	containing
Ctrl	Control
DA	Data Analysis
DEC	Danielle E Carr
DFSA	Drug-Facilitated Sexual Assault
DMP	Dextromethorphan
DRC	Danielle Ross-Carr
DUID	Driving Under the Influence of Drugs
E	empty
Ee	evidence envelope
ELISA	Enzyme Linked Immunosorbent Assay
EtOH	Ethanol, Ethyl Alcohol
Exp.	Expected
Gb	glass bottle
GC	Gas Chromatograph/Gas Chromatography
Gcap	gray capped vial
GCMS	Gas Chromatograph/Mass Spectrometer
GDM	Garry D Metcalfe
Gldcap	gold capped vial
Gsc	Green screw cap
Gvl	gray stopper vial
Gvls	Gray stopper vials
HC Ldr	Hydrocarbon ladder
HPC	high positive control
Htsld	heat sealed
IA	Independent Analysis
Inj.	Injection
Int Std, ISTD, IS	Internal Standard
IPA	Iso-propanol
JSH	Jeff S Hickerson
KAH	Kaitlyn A Hickey; Kait A Hickey
KLH	Kate L Harty
Lavcap	lavender capped vial
Lbcap	light blue capped vial
Lbld	labeled
Lbv	light blue stopper vial
LCL	Lower Control Limit
LCMS	Liquid Chromatography/Mass Spectrometry
LCMSMS	Liquid Chromatography/ Tandem Mass Spectrometry
Lib.	Library
LIMS	Laboratory Information Management System
LLE	Liquid/Liquid Extraction

LLOQ	Lower Limit of Quantitation
LOD	Limit of Detection
LOQ	Limit of Quantitation
LPC	low positive control
LS	Liliana Scifo; Lili Scifo
MAMP	Methamphetamine
MDH	Misti D Hix
MDONE	Methadone
MeOH	Methanol
Mlb	milliliters of blood
Mlsr	milliliters of serum
Mlu	milliliters of urine
Mlwb	milliliters of whole blood
MNB	Melissa N Brous
MNC	Melissa N Cavazos
MNW	Melissa N Windham
MPB	Meprobamate
MRM	Multiple Reaction Monitoring
MSD	Mass Selective Detector
NC	Negative Control
Neg	Negative
Obs.	Observed
OPDS	Opioids
OPIAT	Opiates
Osld	one sealed
Oxyc/M	Oxycodone/Oxymorphone
Pb	plastic bag
Pbt	plastic bottle
Pbts	plastic bottles
PC	Positive Control
PCP	Phencyclidine
Pos	Positive
Pv	plastic vial
QC	Quality Control
qs	Add Quantity Sufficient to Bring Up to Volume
qual	Qualitative
quant	Quantitation
R:, rec'd, rcvd	Received
Rdcap	Red cap
rd/gv	red/gray stopper vial
Rdvl	Red stopper vial
RI	Retention Index
RRT	Relative Retention Time
RT	Retention Time

Saosbi	One sealed "OKLAHOMA STATE BUREAU OF INVESTIGATION Drug Facilitated Sexual Assault BLOOD AND URINE SPECIMEN COLLECTION KIT" containing
Satpd	One sealed "TULSA POLICE DEPARTMENT Drug Facilitated Sexual Assault BLOOD AND URINE SPECIMEN COLLECTION KIT" containing
SD	Standard Deviation
SIM	Selected Ion Monitoring
Sld	sealed
SLM	Sean L Mize
Std Dev	Standard Deviation
STDS	Standards
Stv	one screw top vial
T	trace
TCA	Tricyclic Antidepressants
TE	Tune Evaluation
TIC	Total Ion Chromatogram
TLA	Torrance L Anderson
Tox, TX	Toxicology
TRM	Tramadol
UCL	Upper Control Limit
Ucl	urine specimen collection cup
Ucont	urine container (for anything that is different than DFSA urine specimen cup)
ULOL	Upper Limit of Linearity
ULOQ	Upper Limit of Quantitation
Unksr	# mL of a light red liquid and # mL of a dark red liquid separated by a gel layer. (if unknown vial is serum)
Urn	urine
Usld	unsealed
Vol	Volume
w/	with
WB	Whole blood
WL	Worklist
Ws	Worksheet
ZOL	Zolpidem

Previous Criminalists/Technicians	
Abbreviations	**Definitions**
ABC	Arden B Cavitt
ABH	Arden B Huckeba
JKC	Jerry K Carter
JRM	Janelle R Matthews
KAF	Kayla A Freeman
KEH	Kourtney E Heard
MES	Matt E Stillwell

MPW	M Paul Wallace
RGW	Robert G Weston
SMC	Samantha M Campenni-Hunt

9. ATTACHMENTS

OSBI TOX QMA 2, Rev. 1, Toxicology Drug Standards

OSBI TOX QMA 4, Rev. 0, Drug Receipt and Usage Log

OSBI TOX QMA 10, Rev. 0, Standard Weights Log

10. APPROVAL

FTU Technical Manager — Melissa Brous — Date: 10/27/25

Melissa Brous

CSD Quality Manager — Danielle Ross-Carr — Date: 10/28/25

Danielle Ross-Carr

CSD Director — Janice Joslin — Date: 10/28/2025

Janice Joslin

11. HISTORY

Effective Date	Revision No.	Revised By	Revision Description
11/01/2025	23	M. Brous	Removed history form revision 22. Available in revision 22. Grammatical edits throughout Changed “tubes” to “vials” throughout document In paragraph before scope, I change “the Supervisor” to “their Supervisor”. 2 – Changed “ANAB ISO/IEC 17025:2017Forensic Science Testing Laboratories Accreditation Requirements” to “ANAB Accreditation Requirements for Forensic Testing and Calibration”, removed “Science” and “Subcommittee” from Forensic Science Toxicology Subcommittee 5 – removed “In certain circumstances, the quantification of drugs may be necessary to determine the amount of substance present.” 5.1 – Added “or that were negative” to “If drugs not screened for by immunoassay are identified during the confirmation procedure,…” and “If drugs not screened for by immunoassay are identified during mass spectrometry testing,…” and removed “, and quantitations other than ethyl alcohol” from 3rd paragraph from the end and added “and” in front of fentanyl. Removed “.II.A” after OSBI CSD Quality Manual QP 4. 5.2.1 – Added “or that were negative” to “If drugs not screened for by immunoassay are identified during the confirmation procedure,…” and “If drugs not screened for by immunoassay are identified during mass spectrometry testing,…” and removed “, and quantitations other than ethyl alcohol” from 3rd paragraph from the end and added “and” in front of fentanyl. Removed “.II.A” after OSBI CSD Quality Manual QP 4. Changed “Figure 2” to “Figure 1” in last paragraph and removed “Figure 1 shows the standard case flow for DFSA cases.” 6.5.2 – Removed “Pipette Verification using Artel System The pipettes may be checked using the Artel system instead of the balance as long as all necessary solutions are available. A verification check plan is set on the Artel system. The plan will require five measurements. The CV and accuracy requirements listed for the pipette verification using the balance also apply to the pipette verification using the Artel system. Directions for use of the Artel system are available in the “Toxicology_Lab”

folder on the server." Changed "section 6.5.1" to "Calibration of Analytical Equipment".
6.5.5 – Removed "Artel System" section which reformatted the numbering.
6.5.6 – Capitalized "s" for Standards heading
6.6.3 – Changed "OSBI CSD Quality Manual QP 6.4" to "Physical Evidence Quality Procedures Manual PE QP 2.4". Changed "OSBI CSD QPA 6.4.1" to "Physical Evidence Quality Procedures Manual PE QPA 2.4.1". Added "assigned to refrigerators," in the last paragraph.
6.7.3 – Added "FTU SOP Manual" before TX04 and "ELISA Drug Screen" after in the first sentence.
6.7.4 – Changed "6.7.6.1" to "section Autotune and Tune Evaluation".
6.7.6 – Changed "6.7.6.2" to "Maintenance section".
6.7.6.1 9) – Changed "080821DRC" to "080825MNB".
7, 7.1, 7.2.1, Table 2 LOD, 7.2.8, 7.6.7, 7.6.8, 7.7.7, 7.8.5 – Changed "customers" to "stakeholder".
7.2.1 Table 2. Carryover – Added "internal standard" next to "IS".
7.2.1 – Added "FTU" in front of TM in last sentence of the first bullet point.
7.4.1 – Changed "OSBI CSD Quality Manual QP 5" to "Physical Evidence Quality Procedures Manual PE QP 1". Changed "OSBI CSD Quality Manual QP 6.1" to "Physical Evidence Quality Procedures Manual PE QP 2.1" in two spots.
7.4.2 – Added "can be written: "One sealed OSBI Blood Specimen Collection Kit, blood kit number ###### containing three unsealed 4 mL gray top vials containing whole blood."", changed "The kit" to "An evidence envelope", and added "can be written: "Item 1: One sealed evidence envelope labeled "John Doe" containing items 1A-1B. Item 1A: Two unsealed gray stopper vials containing blood. Item 1B: One unsealed red stopper vial containing blood."".
7.4.5 – Changed "criminalist" to "analyst" before initials throughout. Changed "OSBI CSD Quality Manual QP 6.1" to "Physical Evidence Quality Procedures Manual PE QP 2.1".
7.4.6 – Changed "criminalist" to "analyst", added "in the case narrative", removed "an" in the last sentence.
7.4.8 – Changed "evidence receiving" to "physical evidence units".

7.4.9 – added “at least” in front of 60 days and changed “submitting” to “requesting” agency in the last paragraph.
7.4.10 – Changed “District Attorney” to “Prosecutor”.
7.5.3 – Changed “OSBI CSD Quality Manual QP 7” to “Physical Evidence Quality Procedures Manual PE QP 3”.
7.5.4 – Added “OSBI FTU Quality Manual” in front of 4.2 and changed “DRC 8/14/19” to “MNB 8/14/25” and case sample year form “19” to “25”
7.6.6 – Changed “uc(y)” to “$u_c(y)$”
7.6.11 – Added “mean” subscripted by RSD.
7.7.3 – Removed “For any GC/MS or LC/MS quantitation procedure, the results of the positive control will be entered into the corresponding QC log in Excel which will plot the value. The value(s) should be documented after the calibration run and will be documented before administrative and technical reviews.”
7.7.4 – Added “, hospital vials with the same collection time regardless of cap color” to e.g. and “Confirmation of dextromethorphan in two different hospital vials, same or different colored tops, collected at the same time, by GC/MS or LC/MS-MS analysis.” In the examples.
Removed sections Quantitative GC/MS Identification Criteria and Quantitative LC/MS Identification Criteria in 7.7.4.
7.7.4.2 – Changed “Drug Identification Criteria” to “Drug Presumptive Criteria”, removed “should not be used for identification or reporting purposes and”, Changed NOTE “If a very negative result is observed for a case..” to “If a result more negative than the negative control is observed..”
7.7.4.4 – Added “RI” after retention indices.
7.7.4.5 – changed “reporting” to “of interest(s)”.
7.7.5 – Added an “a” between include and scan in the last sentence of the first paragraph.
7.7.7 – Changed “their own” to “each case” in the first sentence, changed “paperwork” to “documentation” in 5th bullet point, “is” to “are” in 7th bullet point, “them and the report” to “results from each as well as the reported results” in 8th bullet point, removed “statistics are completed and” in 9th bullet point, added “nonconformance” after Class II and changed “established” to “identified” in 2nd paragraph, changed “fix” to“nonconformance correction” before Class II in 3), “D. Ross-Carr” to “M. Brous” in the example of Class II nonconformance.

7.8.3 – Removed “for the subject” in 3rd paragraph.
7.8.3, 7.8.4, 7.8.6, & 7.8.7 – Changed “should” to “shall” for adding no drugs identified report summary clarification.
7.8.5 – Corrected the spelling of “benzoyleconine” to “benzoylecgonine”.
7.8.6 – Removed the Quantitative results section.
7.8.8 2) – added “equal to or” in front of greater than five.
7.8.10 – Changed “their testimony to support a particular sie of a case” to “their testimony beyond the scope of their authorization to work”.
7.8.12 – Took out “The OSBI will perform quantitative analysis on a case-by-case basis and only in cases in which a, b, and c above are met.” And replaced it with “The OSBI FTU does not perform quantitative analysis other than for ethanol.”
Abbreviations – Added CMH for Cortney M Hanna, KLH for Kate L Harty, MNC for Melissa N Cavazos, changed ASB from “American Academy of Forensic Sciences Standards Board” to “Academy Standards Board” removed ASCLD/LAB, made another table labeled “Previous Criminalist/Technicians” and moved analysts not currently in the Toxicology unit to that table.
Updated Toxicology Drug Standards Form by removing “Quantitation Drugs” and color formatting

Toxicology Drug Standards

Drug Standard	Location	Manufacturer/ Preparer	Lot #	Exp. Date	Date Prepared	Date Discarded	Discarded by

Toxicology Drug Reference Material Receipt and Usage Log

Drug: _____

Supplier/Manufacturer/Source: _____

Lot #: _____ Date of Original Receipt: _____

Literary Reference used to verify Reference Material: _____

Date Reference Material was expended and container discarded: By:

Date	Amount Removed	Wt/Vol Remaining	User's Name /Purpose of Standard	User's DEA #

THIS IS A PERMANENT RECORD – DO NOT DESTROY

Oklahoma State Bureau of Investigation
Standard Weights Log
Toxicology Laboratory

Location: _____
Balance: _____
OSBI #: _____

Date	Initials	500g	100g	20g	5g	1g	500mg	100mg	50mg	Recalibrated Y/N

Location: _____
Balance: _____
OSBI #: _____

Date	Initials	500mg	100mg	50mg	20mg	10mg				Recalibrated Y/N