Chemistry Unit
Case Assignment and Evidence Handling Procedures

1 Purpose

This document supplements the practices set forth in the *FBI Laboratory Quality Assurance Manual* and the *FBI Laboratory Operations Manual* by incorporating Chemistry Unit (CU) specific information and requirements.

2 Scope

These procedures apply to CU personnel conducting case assignments and handling evidence in all disciplines/categories of testing with the exception of Fire Debris.

3 Procedures

3.1 Case Assignments

The assignment of cases to CU examiners is the responsibility of the CU Chief, applicable Technical Leader, or applicable Supervisor. When a case is assigned to an examiner, the following steps are taken:

- The request is reviewed to determine which CU discipline(s) and/or category(ies) of testing is/are involved.
- If a new examiner assignment is required, the CU Chief or applicable Technical Leader will ensure the assignment is made to the applicable Case Record in FA. For Legacy cases, the appropriate Evidence Management Unit personnel will be notified of the examiner assignment.

3.2 Evidence Inventory

For Single Unit Submissions, refer to the *FBI Laboratory Practices for Processing a Single Unit Submission (SUS).*

After a case is assigned and the evidence is delivered to the CU, the evidence container(s) and/or packaging will be opened and the contents inventoried. The *CU Evidence Check-In Sheet* (Appendix A) or the FBI Laboratory Evidence Check-In Notes (accessed in FA) will be used for recording the inventory. The evidence listing section of the *CU Evidence Check-In Sheet* is

optional.

A *CU Evidence Check-In Sheet* is not required when the evidence management personnel's check-in notes adequately describe the details of the received evidence, so long as the applicable examiner or technician records that the check-in notes adequately described the evidence as received. If minor edits to the evidence management personnel's check-in notes are necessary, the edits can be made by hand on a printed version of the FBI Laboratory Evidence Check-In Notes.

The *CU Abbreviations List* (Appendix B) contains a list of abbreviations that are not expected to be readily recognized within the field of chemistry, or in general everyday usage. Any other abbreviations that are not expected to be readily recognized need to be defined upon first use within each case file. Abbreviations that are expected to be readily recognized may be used without defining them.

When opening evidence container(s) and/or packaging, CU personnel should keep in mind there may be unsealed items of evidence present. If loose items are found, CU personnel will collect and identify the evidence per FBI Laboratory practices.

3.2.1 Contributor Contact

After inventory and preliminary assessment of the evidence in CU, someone from CU (typically the assigned examiner) will contact the contributor. This communication will be recorded on the Case Communication Log in FA. The contact will allow for communication of matters such as: case investigative needs; time constraints, such as trial dates; clarification on what is forensically feasible and probative; whether additional evidence, such as known samples or reference samples, is required; prioritization of the items to be analyzed; a reasonable estimate of the completion date for the applicable CU examinations; and/or whether the examination(s) is still needed.

This communication is not required for TEDAC cases. For SUS cases, this communication may be excluded if the initial acknowledgement communication is deemed sufficient.

3.2.2 Secondary Evidence

Secondary evidence is material derived from an examination process on an item of evidence (e.g., prepared microscope slides, pill boxes containing debris, vials containing extracts). If sufficient original evidence remains after the examination process such that the process could readily be repeated, then the material is not required to be retained as secondary evidence.

Secondary evidence is recorded on the *CU Secondary Evidence Log* (Appendix C). Refer to the LOM for further information on secondary evidence (transfer, records, etc.).

3.3 Processing and Preservation of Evidence

The processing area and utensils will be appropriately cleaned prior to introducing evidence. Accessory lighting and magnification may be used as needed.

CU caseworking personnel will at all times be aware of the need to protect the evidence for examinations that are to be conducted by other caseworking units, and preserve it from loss, contamination, and deleterious change. These individuals, through their training and by referencing the *Examination Plan*, will be knowledgeable of the order in which examinations need to be conducted. Listed below are general guidelines for evidence preservation. If there are any questions, or if unusual circumstances arise, consult with the other units assigned to the case.

- Perishable Evidence: Perishable evidence (e.g., biological specimens, food items) will be refrigerated or frozen.
- Trace Evidence: Evidence to be examined by the Trace Evidence Unit (TEU) will be opened and scraped by TEU before other examinations are conducted, unless directed otherwise.
- Firearms: If firearms are received, CU caseworking personnel should request assistance of personnel in the Firearms/Toolmarks Unit to render them safe, if such assistance is needed.
- Drug Residue: Evidence involving suspected drug residue will be opened and processed by appropriate CU personnel before other examinations are conducted.
- Ignitable Liquids: Evidence involving suspected ignitable liquids will be opened and processed by appropriate personnel before other examinations are conducted.
- Documents: If indented writing examinations are to be conducted, the technician or examiner will protect the evidence from any action that might impart (or transfer) impressions onto the evidence, including the use of initials to place the evidence under proper seal.
- Latent Fingerprints: Technicians and examiners will preserve latent fingerprint evidence by wearing nitrile or cotton gloves when handling the evidence. Cotton gloves absorb moisture and should not be used to handle latent evidence with non-porous surfaces. Further, examinations that may obliterate possible latent fingerprints should be limited until the fingerprint examinations are completed. If possible, avoid refrigerating latent fingerprint evidence.
- DNA Evidence: Evidence to be examined for DNA should be handled carefully to prevent addition and/or loss of DNA. The use of appropriate personal protective equipment (e.g., lab coat, gloves, and a mask) minimizes the chance of transferring DNA to the evidence.

3.3.1 Autosampler Verification

When an autosampler is used, a sequence log containing the file name, autosampler position, and sample identification will be printed, or otherwise retained. For instruments that do not have the ability to print a sequence log, or in other situations when a sequence log cannot be obtained, the *CU Autosampler Verification Log* (Appendix D) will be completed and retained. The sequence log or *CU Autosampler Verification Log* will be completed by the instrument operator and will be initialed by the operator to indicate that the sequence was checked against the sample position(s) to ensure the two are in agreement.

3.4 Evidence Seal and Storage

Refer to the *FBI Laboratory Quality Assurance Manual* and *FBI Laboratory Operations Manual* for evidence seal and storage requirements; the below sections address CU specifics.

Other than the below exceptions, evidence will be stored in an Evidence Storage Room (ESR) Redacted at the end of each day. When evidence is stored in a location other than an individually assigned locker within an ESR, each transfer to-and-from the storage location (e.g., cage, refrigerator, shelf) will be recorded in FA, or on a *Chain-of-Custody Log* for Legacy cases.

Evidence that is not transferred to an ESR at the end of the day will be secured by placing an "Evidence Do Not Disturb" sign (or similar) on top or in front of the evidence and locking the door to the room (where possible). This practice is limited to evidence that is too large and/or bulky to transfer, or evidence that is being processed in a manner that prohibits transfer (e.g., mounted in equipment). Transfer of the evidence to the non-ESR storage location will be recorded at the end of each day it was examined.

3.4.1 Active Examination

Evidence is considered to be under active examination when it is in the process of being inventoried or examined in the CU. If a period of ten business days elapses since the last time the evidence was inventoried or examined, then the evidence will be considered 'not under active examination' at that point.

3.4.2 Evidence Seal- Boxes With Zip Tie Style Closures

Evidence will be sealed according to the LOM. One additional option is the use of boxes with zip tie style closures. These boxes will be considered properly sealed when zip ties are applied to both ends of the box and each zip tie is initialed by the individual sealing the box.

3.5 Repackaging Drug Evidence Following CU Exams

Prior to returning evidence, any item(s) that require additional examinations (e.g., latent prints) will be separated from bulk drug evidence and repackaged in a new plastic evidence envelope, or an alternative package according to the LOM. A completed *FBI Laboratory Drug Evidence Label* (7-248) will be affixed to the package and the package will be properly sealed. The bulk drug evidence will be repackaged and properly sealed according to the LOM.

4 References

FBI Laboratory Quality Assurance Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

FBI Laboratory Operations Manual, Federal Bureau of Investigation, Laboratory Division, latest revision.

FBI Laboratory General Description of Evidence, Federal Bureau of Investigation, Laboratory Division, latest revision.

Forensic Advantage User Guide, Forensic Advantage® Systems, a division of The Computer Solution Company, Inc., latest revision.

Rev. #	Issue Date	History
9	02/09/18	Removed "policies" from section 1. Minor changes to section 2 for clarity and to meet new scope requirements. Changed "Examiner" to "examiner"; "Chemist/Physical Scientist" to "technician"; "evidence analyst" and "request coordinator" to "evidence management personnel" throughout. Changed "document" to "record" (and similar) throughout. Changed "Subunit Manager" to "Technical Leader" throughout. Removed "or designee" in section 3.2, added "applicable". Edited sections 3.3, 3.3.2, 3.4.1 to account for changes in forms (see following Appendices edits, to include consolidation to one CU Check-In Sheet version) and removed reference to locations of forms since same as any controlled form (i.e., BUNET and LABNET), added FBI Check-In-Notes (in FA) as an option. Added reference to SUS practice in section 3.3. Added option of minor handwritten edits to EA's/RC's printed check-in notes in section 3.3. Added section 3.3.1 (similar language to previous LOM requirement). Minor edits to secondary evidence descriptions in section 3.3.2. Added reference to GDE in section 3.3. Added reference to *Examination Plan* and removed footnote on cotton gloves in section 3.4 (embedded in section), added Ignitable Liquids to section 3.4. Removed previous section 3.5 (transfers of evidence for instrumental analysis). Revised new section 3.5 for clarity and added additional requirements, to include storage in an ESR each day and recording of transfers to non-personal storage locations. Removed reference to FA labels/barcodes and reference to *FBI Laboratory Practices for Handling Drug and Valuable Evidence* in section 3.6. Deleted previous Appendix A (3 page check-in form) and replaced with previous Appendix B ('alternate version'). Previous Appendix C (abbreviations) became Appendix B, added "NCS" and "PCS" to list, removed "PB" as abbreviation for "pillbox", and removed "PCH" as "positive control hair". Previous Appendix D (secondary evidence log) became Appendix C. Deleted previous Appendix E (handwritten secondary evidence log). Previous Appendix F (autosampler log) became Appendix D.
10	09/13/19	Removed previous section 3.1 since hand-to-hand transfer of drug and valuable evidence is no longer required and rest of content was unnecessary, renumbered remaining sections. Revised to "Evidence Management Unit" in section 3.1. Changed "FBI Check-In-Notes" to FBI Laboratory Evidence Check-In Notes in section 3.2. Changed section 3.2.1 to allow the initial acknowledgement communication to be deemed sufficient for SUS cases. Edited

section 3.4 to include the new FA bulky storage locations and to clarify the recording of internal transfers. Updated drug label name in section 3.5. Removed previous section 3.5.3 since drug and valuable evidence will no longer need to be stored in separate lockers.

Approval

Redacted - Signatures on File

General Chemistry
Technical Leader: Date: 09/11/2019

Acting Toxicology
Technical Leader: Date: 09/11/2019

Metallurgy
Technical Leader: Date: 09/11/2019

Paints and Polymers
Technical Leader: Date: 09/11/2019

Chemistry Unit Chief: Date: 09/11/2019

QA Approval

Quality Manager: Date: 09/11/2019

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Appendix B: *CU Abbreviation List*

ABS	acrylonitrile-butadiene-styrene co-polymer
BOPP	biaxially-oriented polypropylene
bpt	black plastic tape
c, c/	containing or with
cap	capsule
CD, c.d.	cross direction
CT	culture tube
gms	glass microscope slide
gws	glass well slide
GWt	gross weight
HDPE	high-density polyethylene
HS	heat-sealed
HSB	heat-sealed bag
HSE	heat-sealed envelope
HSEE	heat-sealed evidence envelope
LDPE	low-density polyethylene
LM	left message
MD, m.d.	machine direction
MMA	methyl methacrylate
MMY	make/model/year
MOPP	monoaxially-oriented polypropylene
N/C	no change
NC	negative control
NCB	negative control blood
NCS	negative control serum
ND, n.d.	not detected
N/R	no reaction
NR, n.r.	not reporting
OEM	original equipment manufacturer
PB	plastic bag
PBX	pill box
PC	positive control
PCB	positive control blood
PCH	positive control high
PCL	positive control low
PCS	positive control serum
PCU	positive control urine
PE	polyethylene
PP	polypropylene
PS	polystyrene
SBR	styrene butadiene rubber
SIS	styrene-isoprene co-polymer
tab	tablet
TM	test mix
TT	test tube
TWt	tare weight
VCF	vacuum collection filter
w/f	warp and fill
ZPB, ZPLB	Ziplock/zippered plastic bag

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Chemistry Unit
Open Proficiency Testing Procedures

1 Purpose

This document describes the procedures for the internal and external proficiency testing program of the Chemistry Unit (CU), in compliance with the *FBI Laboratory Practices for Open Proficiency Testing*.

2 Scope

The CU Proficiency Testing Program encompasses both internal and external proficiency testing. Qualified personnel within the CU who perform casework in the categories of testing listed within this document are subject to the procedures contained herein.

3 Frequency of Proficiency Tests

Each qualified examiner and technician must complete one annual open proficiency test in each "Test Description" listed in the table (Appendix A) in which he/she routinely performs casework. This testing will cover the procedures the participant routinely uses in casework. Additionally, if the CU Chief chooses to remain proficient in his/her respective category(ies) of testing, he/she will participate in the applicable proficiency test(s) to the extent that he/she would perform procedures in casework.

4 Distribution

Prior to distribution of any proficiency test, the "Participant & Test Info" and "Test Preparation" sections of the *CU Open Proficiency Test Preparation and Evaluation Form* (Appendix B) will be completed.

The CU proficiency test representative (CU PTR) will ensure the proficiency test and associated paperwork is delivered to the test participant. The test participant will sign and date an acknowledgment receipt of the proficiency test. This receipt will be kept in the proficiency test file for that test participant.

5 Division of Labor for Analyses Conducted on Proficiency Tests

Each technician will perform the analytical portion of his/her proficiency test after consultation with an assigned examiner if necessary. The technician will perform a complete range of analytical work on the proficiency test commensurate to the extent he/she performs casework.

Each examiner will complete his/her proficiency test independently. The only exceptions are the Forensic Toxicology (see section 7.1.2), Toxicology Survey (see section 7.1.3), and Drug Facilitated Crime (see section 7.1.4) tests.

6 Internal Proficiency Tests

6.1 Preparation of Internal Proficiency Tests

The following internal proficiency tests will be prepared as needed in the CU by the CU PTR and/or an examiner/technician that is qualified in the applicable category of testing. Any other personnel participating in sample preparation will need prior approval from the applicable Technical Leader. This approval will be recorded on the *CU Open Proficiency Test Preparation and Evaluation Form* or attached records.

- Bank Security Chemicals
- Metallurgy (Trace Metal Comparison)

All internal proficiency tests and samples for these tests will be prepared and recorded following the *FBI Laboratory Practices for Open Proficiency Testing*. Sample and test preparation records will be retained permanently in the proficiency test file.

6.2 Test Design

The test designs for the CU's proficiency tests are detailed as follows.

6.2.1 Bank Security Chemicals

Bank security devices consist of money packs containing 1-methylaminoanthraquinone (MAAQ), while some packs also contain orthochlorobenzalmalononitrile (CS tear gas). An acceptable proficiency test entails correct identification of the presence or absence of MAAQ and/or CS tear gas.

6.2.1.1 Internal Proficiency Test Preparation for Bank Security Chemicals

The internal proficiency test for bank security chemicals will consist of either a positive or negative test. The sample(s) of material will be placed in appropriate packaging and given a unique identifier(s). The sample(s) and test preparation information will be provided to the CU PTR. The CU PTR will ensure the test sample(s) is distributed to the participant.

6.2.1.1.1 Positive Internal Proficiency Test for Bank Security Chemicals

The positive internal proficiency test for bank security chemicals will consist of a detectable amount of known MAAQ standard deposited on a suitable substrate (e.g., cloth swatch). CS tear gas may also be deposited on the substrate.

6.2.1.1.2 Negative Internal Proficiency Test for Bank Security Chemicals

The negative internal proficiency test for bank security chemicals will consist of a red stain similar in color to MAAQ (e.g., food coloring, lipstick, felt tip ink, or a red colored beverage) deposited on a suitable substrate (e.g., cloth swatch). No MAAQ or CS tear gas will be added.

6.2.2 Metallurgy (Trace Metal Comparison)

A wide variety of metals are submitted to the CU for analysis. An examination may be requested to determine the composition of the material and/or to determine if source commonality exists.

6.2.2.1 Internal Proficiency Test Preparation for Metallurgy

The internal proficiency test for metallurgy (i.e., trace metal comparison) will consist of either a positive or negative association based on the composition of the material. All samples provided to the participant will come from a source that has an established composition. The sources include National Institute of Standards and Technology (NIST)-traceable standards or metal samples of known provenance. The samples should be chosen to be homogeneous for the purposes of analysis and should be of sufficient size to allow all necessary analyses to be performed. The individual samples will be placed in appropriate, separate packaging and given unique identifiers. The sample(s) and test preparation information will be provided to the CU PTR. The CU PTR will distribute the test sample(s) to the participant.

6.2.2.1.1 Positive Internal Proficiency Test for Metallurgy

The positive internal proficiency test for metallurgy will consist of a minimum of three splits of samples provided to each participant. At least two of the splits will be from the same source and the remaining split(s) may be from a different source.

6.2.2.1.2 Negative Internal Proficiency Test for Metallurgy

The negative internal proficiency test for metallurgy will consist of a minimum of three splits of samples provided to each participant. All of the splits will be from different sources.

6.3 Record Keeping for Preparation of Internal Proficiency Tests

In addition to retention of sample and test preparation records per the *FBI Laboratory Practices for Open Proficiency Testing*, the "Test Preparation" and the upper right box of the "Participant & Test Info" sections of the *CU Open Proficiency Test Preparation and Evaluation Form* will be completed. Optionally, this information may be attached to the form. The identification of the samples included in the test as well as the expected results (including acceptable limits when appropriate) will be attached to the form.

6.4 Time Limit for Completion of Internal Proficiency Tests

The time limit for completion of all internal proficiency tests in the CU is six weeks. The due date will be included in a memo accompanying the proficiency test and on the *CU Open Proficiency Test Preparation and Evaluation Form*. An extension of this due date will not be granted unless a written request has been made to the CU Chief at least 3 days prior to the due date and that request has received appropriate approval.

7 External Proficiency Tests

The CU PTR will ensure the following external proficiency tests are procured for the CU.

- From Collaborative Testing Services, Inc.
  - Controlled Substances Analysis
  - Ignitable Liquid Identification
  - Paint Analysis

- From College of American Pathologists (CAP)
  - Whole Blood Alcohol/Ethylene Glycol/Volatiles (Survey AL1)
  - Forensic Toxicology (Survey FTC)
  - Toxicology Survey (T-Series)
  - Drug Facilitated Crime (Survey DFC)

- From Forensic Testing Services
  - Tape Analysis

- From ASTM International
    - Metallurgy (Steel Quantitation)

Generally, these proficiency tests are administered as directed by the external provider.

7.1 External Proficiency Test for Fire Debris Analysis

Fire Debris proficiency tests involve the qualitative analysis of items for the presence of ignitable liquid or ignitable liquid residues. Each qualified examiner and technician conducting fire debris analysis will take one ignitable liquid identification proficiency test per year.

7.2 External Proficiency Test for Tape Analysis

In the event that the test sample(s) received consist of a type of tape not routinely analyzed by CU (e.g., office tape, masking tape) Paints and Polymers personnel will prepare an internal proficiency test using retained external proficiency test samples (refer to the *FBI Laboratory Practices for Open Proficiency Testing*).

7.3 External Proficiency Tests for Toxicology

Toxicology proficiency tests involve the qualitative and potentially quantitative analysis of biological samples for the presence of drugs and other potentially toxic substances. Each qualified examiner and technician working in the Toxicology team will take at least one Whole Blood Alcohol/Ethylene Glycol/Volatiles (Survey AL1) proficiency test per year. Each examiner and technician may assist in the analysis of the Forensic Toxicology (Survey FTC), Toxicology Survey (T-Series), and Drug Facilitated Crime (Survey DFC) tests, but this will not count as an individual proficiency test.

7.3.1 Whole Blood Alcohol/Ethylene Glycol/Volatiles (Survey AL1)

This test is designed for laboratories that perform analysis for ethanol and other volatiles in whole blood specimens for clinical or forensic purposes. Since the Toxicology team is fully validated for ethanol, methanol, acetone, isopropanol, and ethylene glycol, these volatiles must be quantitated if identified within a sample. A subscription to Survey AL1 provides three tests per year that are shipped at different times. Each test consists of five liquid whole blood specimens. All specimens contained within a single Survey AL1 must be analyzed by the test participant.

7.3.2 Forensic Toxicology (Survey FTC)

This test is designed for laboratories that perform qualitative and quantitative analysis of drugs and metabolites in antemortem and postmortem blood and urine. A subscription to Survey FTC provides two tests per year that are shipped at different times. The tests may include a paired urine and blood sample. The urine and blood samples will be analyzed qualitatively. The blood sample will also be analyzed quantitatively if a validated SOP(s) exists for the analyte(s) detected. All samples within the Survey FTC will be analyzed by the Toxicology team in order for the survey to be considered as complete. Each test will be assigned to one examiner who will coordinate the analysis of the samples, collect all data, and assemble the final results.

7.3.3 Toxicology Survey (T-Series)

In order to meet current American Board of Forensic Toxicology (ABFT) requirements, the Toxicology team will take this test in addition to the rest of the required toxicology proficiency tests. A subscription to the T-Series provides three tests per year that are shipped at different times. Each test may include serum and urine samples, some of which may be paired. The urine and serum samples will be analyzed qualitatively. The serum sample(s) will also be analyzed quantitatively if a quantitation is being performed on casework samples while the proficiency test is in house. Each test will be assigned to one examiner who will coordinate the analysis of the samples, collect all data, and assemble the final results.

7.3.4 Drug Facilitated Crime (Survey DFC)

The Toxicology team will take this test in addition to the rest of the required toxicology proficiency tests. A subscription to the Survey DFC provides two tests per year that are shipped at different times. The tests consist of urine samples that will be analyzed qualitatively. Each test will be assigned to one examiner who will coordinate the analysis of the samples, collect all data, and assemble the final results.

8 Reporting and Evaluation of Proficiency Test Results

Proficiency test results will be evaluated and reported following practices established by the *FBI Laboratory Practices for Open Proficiency Testing* and, when applicable, the external test provider. The CU Test ID # will be recorded on each administrative and examination record.

8.1 Division of Labor for Reporting Results

For a proficiency test assigned to a technician, the technician will provide all notes and analytical data to an examiner qualified in the applicable category of testing. For internal proficiency tests, the technician and examiner will complete their respective portions of the *CU Internal*

Proficiency Test Results Form (Appendix C). For external proficiency tests, the examiner will complete the external provider results datasheets.

8.2 Evaluation of Completed Proficiency Tests

The "Evaluation" section of the *CU Open Proficiency Test Preparation and Evaluation Form* will be completed by the CU PTR for each proficiency test administered, with the exception of his or her own proficiency test(s) which will be evaluated by the CU Chief. The Evaluator, CU Chief, and Participant will then sign and date the form after reviewing the proficiency test and summary report(s).

8.3 Additional Reporting

The CU PTR will forward a copy of all CAP Participant Surveys and Evaluations results to the Toxicology Technical Leader for review and filing purposes.

9 Completed Proficiency Test Records

The CU Chief will ensure that the relevant records associated with a completed proficiency test are maintained permanently. The following records will be maintained in the CU and/or in FA:

- *CU Open Proficiency Test Preparation and Evaluation Form*
- Administrative and examination records
- Results of test and sample validations (for internal proficiency tests)
- Memo from the CU PTR to the test participant that accompanies the proficiency test, including signed acknowledgement for receipt of the proficiency test
- *CU Internal Proficiency Test Results Form*
- Results and evaluation notices from the supplier of a purchased test
- All notices to and from the Laboratory Division Proficiency Test Program Manager (PTPM) concerning a particular test
- Original datasheets, or printed copies of electronic datasheets, submitted to external proficiency test providers

10 Corrective Action

In the event that a proficiency test results in an error requiring corrective action, the CU Chief will follow the practices described in the *FBI Laboratory Practices for Open Proficiency Testing*.

11 Completed Proficiency Test Samples

Upon completion of a proficiency test, all proficiency test samples will be appropriately packaged and returned to the CU PTR. The CU PTR will retain the samples until the associated results have been received and evaluated and any corrective actions associated with that test have been resolved.

Rev. #	Issue Date	History
12	09/13/19	Changed "practices" to "procedures" in section 2. Removed last sentence in section 2 since non-CU personnel supporting CU casework (e.g., technical reviewers) are no longer required to take proficiency tests. Removed section 6.2.2 ("Polymeric Materials"- external tape proficiency test fulfills 4.15 category of testing), renumbered remaining sections and edited list in section 6.1. Added section 7.1 and renumbered remaining sections. Defined "PTPM" acronym in section 9. Revised Appendices A and B for consistency with ANAB Categories of Testing and to reflect removal of Polymeric Materials internal test. Simplified Appendix B Evaluation section.
13	12/02/19	Added Ignitable Liquid Identification to Section 7 listing of external tests. Added explanation of Fire Debris proficiency test as Section 7.1. Added approval signature line for Fire Debris Technical Leader. Appendix A updated to include Category of Testing 4.10 Fire Debris. Appendix B updated to include Fire Debris.

Approval

Redacted - Signatures on File

General Chemistry
Technical Leader: Date: 11/29/2019

Acting Toxicology
Technical Leader: Date: 11/29/2019

Metallurgy
Technical Leader: Date: 11/29/2019

Fire Debris
Technical Leader: Date: 11/29/2019

Paints and Polymers
Technical Leader: Date: 11/29/2019

Chemistry Unit Chief: Date: 11/29/2019

Redacted - Signatures on File

QA Approval

Quality Manager: Date: 11/29/2019

Appendix A: *CU Proficiency Tests*

Discipline	Category(s) of Testing	Test Description	Frequency	Source
1.0 Drug Chemistry	1.1 Controlled Substances, 1.3 General Chemical Testing	Drugs	1/year/individual	External
2.0 Toxicology	2.1 Human Performance Forensic Toxicology, 2.2 Post-mortem Forensic Toxicology, 2.3 General Forensic Toxicology	Whole Blood Alcohol/Ethylene Glycol/Volatiles (Survey AL1)	1/year/individual	External
		Forensic Toxicology (Survey FTC)	2 team tests/year	External
		Toxicology Survey (T-Series)	3 team tests/year	External
		Drug Facilitated Crime (Survey DFC)	2 team tests/year	External
4.0 Trace Evidence	4.1 Paint	Paint	1/year/individual	External
4.0 Trace Evidence	4.10 Fire Debris	Ignitable Liquid Identification	1/year/individual	External
4.0 Trace Evidence	4.15 General Physical and Chemical Analysis	Bank Security Chemicals	1/year/individual	Internal
		Tape	1/year/individual	External
		Metallurgy (Trace Metal Comparison)	1/year/individual	Internal
		Metallurgy (Steel Quantitation)	1/year/individual	External

Appendix B: *CU Open Proficiency Test Preparation and Evaluation Form*

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Appendix C: *CU Internal Proficiency Test Results Form*

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Chemistry Unit
Case Record and Review Procedures

1 Purpose

This document describes the Chemistry Unit (CU) procedures for recording and reviewing case-related materials, in compliance with the *FBI Laboratory Quality Assurance Manual* and *FBI Laboratory Operations Manual*.

2 Scope

These procedures apply to CU personnel preparing case records and conducting case reviews.

3 Case Records

The combination of the 1A (or 1C) envelope(s) (*Supporting Documentation Envelope* (7-251)) and electronic files uploaded to Sentinel are referred to as the "case record".

3.1 Administrative Records

Records that do not pertain to the conclusions of the examinations performed are considered administrative records. The FBI Laboratory Number will be on each page of administrative records or on at least the first page of any bound administrative records.

The following are defined as administrative records in the CU:

- Incoming communications
- *FBI Laboratory Chain of Custody* forms (7-243 or 7-243a, Legacy cases)
- Forensic Advantage (FA) Chain of Custody
- *Chemistry Unit Secondary Evidence Log*
- *FBI Laboratory Activity and Communication Log* (7-245, Legacy cases)
- FA Communication Logs
- *FBI Laboratory Work Sheet* (7-2, Legacy cases)
- FA Case Report and FA Case Record Report

3.2 Examination Records

Case notes, forms, printouts, charts, and other records that pertain to the conclusions of the examinations performed are considered examination records.

The FBI Laboratory Number, the date of examination(s), and the examiner's handwritten initials/name/signature (or secure electronic equivalent) will be on each hard copy page of the examination records. An examiner's handwritten initials/name/signature will acknowledge his/her agreement with the content of the examination records.

When examination records are prepared by a technician (or by an examiner performing work as a technician), the preparer's handwritten initials/name/signature (or secure electronic equivalent) will be on each hard copy page of the examination records representing his/her work.

If examination records are maintained only in FA, Laboratory practices will be followed for recording review and agreement of these records.

The following are defined as examination records or case notes in the CU:

- Handwritten or typed notes that describe observations and/or exams conducted [to include Case Notes Interface (CNI) in FA]
- Check-in notes
- Instrument printouts, including operating conditions
- Instrument sequence log (or *CU Autosampler Verification Log*)
- Calculations
- Photographs

3.3 Accounting for Pages in a Case Record

The totality of the administrative and examination records (together or separately) will be accounted for and recorded. The FA Publish and Packet Manager will be used to account for electronic files that are maintained only in FA and then uploaded to Sentinel. One of the following techniques will be used to account for hard copy pages within the CU.

- Number the pages of the administrative and examination records in the form of "Page__ of __".
- Number each page of the administrative and examination records sequentially, indicating the last page in some manner.
- On the 1A envelope, write a description of the type of record and the number of each type present.

4 Case Review

The review of case records encompasses three forms of review: verification of identifications and associations, technical review, and administrative review. Each review process must be completed and recorded prior to issuing a *Laboratory Report* (7-1, 7-1 LIMS, 7-273, or 7-273 LIMS) to a contributor.

An association, as defined by the CU, exists between two or more items if they possess one or more characteristics that indicate they could have originated from a common source. The strength of the association can vary and depends on the characteristics observed. Details on the nature and relative strength of an association between evidentiary items and/or evidentiary item(s) and known materials will be provided in the *Laboratory Report*.

4.1 Technical Reviews and Verifications of Identifications and Associations

In the CU, the technical review and verification of identifications and associations are combined into a single review process and will be conducted in accordance with Laboratory practices. This technical review will include a check of manual calculations, data transcriptions, and data reductions relevant to the examinations.

Another authorized individual in the category of testing must conduct the technical review. The author should verify that the technical reviewer is available to conduct the review and then use FA to request the technical review, or simply provide the technical reviewer with the *Laboratory Report* and supporting records for Legacy cases.

An examiner may perform a technical review on a case record that contains a "packet(s)" of examination records that he/she authored/co-authored, provided that the packet(s) has been technically reviewed by another authorized individual in the category of testing that did not co-author the packet(s) in question. The technical review of the packet(s) will be recorded on the first page of the packet(s) with the reviewing examiner's initials and/or signature, the date, and a statement indicating that the packet(s) has been technically reviewed.

The case record technical review and verification of identifications and associations will be recorded within FA, or on the following line of the *Laboratory Report* file copy for Legacy cases:

Technically reviewed and identifications and associations verified by: _____ Date: _____

4.2 Administrative Reviews

The UC and all qualified CU examiners are authorized to perform administrative reviews. The author of the *Laboratory Report* will use FA to request an administrative review, or simply provide the administrative reviewer with the *Laboratory Report* and supporting records for Legacy cases. The administrative review will be carried out in accordance with Laboratory practices and recorded in FA, or on the following line on the yellow file copy of the *Laboratory Report* for Legacy cases:

Administratively reviewed by: _____ Date: _____

An examiner may perform an administrative review on a case record that contains a "packet(s)" of examination records that he/she authored/co-authored, provided that the packet(s) has been administratively reviewed by another qualified examiner in CU. In this situation, the administrative review of the packet(s) will be recorded on the first page of the packet(s) with the reviewing examiner's initials and/or signature, the date, and a statement indicating that the packet(s) has been administratively reviewed.

5 Expedited Results

Expedited or partial results of an examination(s) may be disseminated prior to issuing a *Laboratory Report*. When opinions and/or interpretations are directly communicated by dialogue with the contributor, a record of the specific dialogue will be retained in the appropriate communication log. See the *FBI Laboratory Quality Assurance Manual, FBI Laboratory Practices for Preparing, Reviewing, and Issuing Laboratory Reports and Retaining Records in Forensic Advantage (FA*), and the *FBI Laboratory Practices for Preparing, Reviewing, and Issuing Laboratory Reports and Retaining Records for Legacy Cases* for further details. The following expedited or partial results of an examination(s) do not need to be verified by another appropriately qualified CU examiner prior to dissemination.

- Negative results
- Presumptive results
- Physical characteristics

6 Alternate Reporting

When reporting on initiatives or intelligence matters, results may be issued via an Electronic Communication (EC, FD-1057). The EC is the approved CU alternate reporting format. Supporting documentation and review requirements for alternately reported results are the same as for a *Laboratory Report*. The Case Record Status will be changed to "Complete" in FA prior to generating the Combined Case and Case Record 1A (or Case Record 1A). The technical and administrative reviews will be recorded by including the reviewers as approvers in Sentinel.

7 Recording and Reviewing Concessions and Corrections

Refer to the *FBI Laboratory Practices for Addressing a Nonconformity* for definitions of concessions and corrections. Concessions and corrections will be recorded within a logbook that is maintained by the Unit Chief. The Unit Chief will review this logbook annually (at a minimum) in an effort to identify any trends. The reviews and any observed trends will be recorded.

Rev. #	Issue Date	History
7	09/13/19	Minor edits to definition of 'examination records' in section 3.2 to align with Level 1 documents. Changed "qualified examiner" to "authorized individual" in section 4.1. Changed Legacy technical review example in section 4.1 to "verified" (was "confirmed"), and removed redundant language in same part of the section. Added "initials and/or" to sections 4.1 and 4.2 for packet reviews. Revised section 4.2 for clarity. Added "specific dialogue" requirement and reference to *FBI Laboratory QAM* to section 5. Added "physical characteristics" to section 5. Added recording of trends to last sentence in section 6.
8	12/02/19	Added section 6 (alternate reporting).

Approval

Redacted - Signatures on File

General Chemistry
Technical Leader: Date: 11/29/2019

Acting Toxicology
Technical Leader: Date: 11/29/2019

Metallurgy
Technical Leader: Date: 11/29/2019

Paints and Polymers
Technical Leader: Date: 11/29/2019

Chemistry Unit Chief: Date: 11/29/2019

QA Approval

Quality Manager: Date: 11/29/2019

Chemistry Unit
Procedures for the Use of Reference Materials and Known Materials

1 Purpose

The Chemistry Unit (CU) uses reference materials for qualitative and quantitative analysis. This document describes the CU's procedures for the acquisition, storage, and verification of reference materials. Certified reference materials do not require verification within the CU. Additionally, Metallurgy maintains a set of metal reference materials that do not require verification. These metal reference materials are accompanied by certificates that justify the scope of their use, however some of the certificates do not meet all of the requirements to allow the materials to be classified as certified reference materials. Refer to the *CU Procedures for Measurement Traceability* for information regarding the establishment of measurement traceability through the use of reference materials.

A known material is an item acquired for the purpose of comparison with an evidentiary sample (e.g., commercial products, items received directly from manufacturers). This differs from a reference material in that only the source of the known material, not the exact composition, needs to be known at the time of acquisition. This document describes the information that will be recorded when using a known material in casework.

2 Scope

This document applies to CU personnel that acquire, store, and/or verify reference materials acquired for use in casework in all disciplines/categories of testing with the exception of Fire Debris. This document also applies to CU personnel that use known materials in casework in all disciplines/categories of testing with the exception of Fire Debris.

3 Equipment/Materials/Reagents

The appropriate equipment, materials, and reagents used to verify a reference material will depend upon the nature (organic/inorganic, polar/nonpolar, etc.) of the substance. Since most of these verifications will be performed following an established CU standard operating procedure, the equipment, materials, and reagents required for such will be listed within the procedure used.

Known materials will undergo the same relevant analytical examinations that are performed on a questioned sample(s) during casework. The equipment, materials, reagents, and other relevant information may be found in the applicable standard operating procedure(s) being used.

4 Procedures

4.1 Purchasing a Reference Material

A *Requisition for Supplies and/or Equipment* (FD-369 or equivalent) will be prepared for all reference materials to be purchased. All FD-369 forms (or equivalent) for reference materials will be approved by the Unit Chief prior to ordering. CU's Chemical Inventory Manager (CIM) will be notified of reference material purchases/receipts through the CU Chemical Products database, or by receiving a copy of the FD-369 (or equivalent) from the CU purchase credit card holder.

When appropriate, a certified reference material will be purchased. When a reference material is received in the CU, the CIM will verify that a Certificate of Analysis (COA) is requested/received from the manufacturer, if available.

4.2 Storage of Reference Materials

The reference material will be stored following manufacturer's recommendations.

4.2.1 Storage of Controlled Substances

If the reference material received is a controlled substance, the initial product weight will be recorded electronically in the CU Chemical Products database.

All controlled substances (with the exception of low concentration solutions, such as 1 mg/mL reference material solutions) will be stored in Redacted , which is an evidence storage room (ESR) secured for dual-person entry. When entering Redacted to acquire a reference material, the *Access Log – Evidence Storage Facility* (FD-455) will be filled out.

4.3 Opening Reference Materials

4.3.1 Opening Controlled Substance Reference Materials

When opening a controlled substance reference material for the first time, it is good laboratory practice to record the date and your initials directly on the container; however, this is not a required practice. Each time any amount of a controlled substance reference material is removed from its container, the before and after weights of the container will be recorded in the CU Chemical Products database.

4.3.2 Opening Non-Controlled Substance Reference Materials

When opening non-controlled substance reference materials for the first time, it is good

laboratory practice to record the date and your initials directly on the container; however, this is not a required practice.

4.4 Synthesis of a Reference Material

When a suitable reference material is not available from a vendor, it may be necessary to synthesize it. The following information will be recorded and provided to the CIM.

- The procedure used to synthesize the material
- The date of synthesis
- The initials of the person who synthesized it
- Any unique storage requirements
- Controlled substance schedule, if applicable

4.5 Reference Material Verification

Certified reference materials do not require verification. Non-certified metal reference materials that are used within the scope defined on their respective certificates do not require verification. For all other non-certified reference materials, only one sample per manufacturer's lot number must be verified. Subsequent reference materials from the same lot will be considered as having the same verification as the original. The identity of the reference material will be verified prior to, or in concurrence with casework. If the reference material is to be used for quantitative work, the purity of the reference material must also be verified prior to, or in concurrence with casework. The following lists the steps necessary to perform verifications.

4.5.1 Identity Verification

Use one or more of the following techniques (as appropriate) to verify the identity of the reference material:

- Gas Chromatography/Mass Spectrometry (GC/MS)
- Solids Probe Mass Spectrometry (SP/MS)
- Pyrolysis Gas Chromatography/Mass Spectrometry (Py-GC/MS)
- Fourier Transform Infrared Spectroscopy (FTIR)
- Raman Spectroscopy
- Liquid Chromatography/Mass Spectrometry (LC/MS)
- High Resolution Mass Spectrometry (e.g., OrbiTrap XL or Direct Analysis in Real Time Time-of-Flight Mass Spectrometry (DART))
- X-ray Powder Diffraction (XRD)
- Scanning Electron Microscopy with Energy Dispersive X-ray Spectrometry (SEM/EDS)

- X-ray Fluorescence Spectroscopy (XRF)
- High Performance Liquid Chromatography (HPLC) with applicable detector(s)
- Gas Chromatography (GC) with applicable detector(s)
- Ultraviolet-Visible Spectroscopy (UV-Vis)
- Inductively Coupled Plasma/Mass Spectrometry (ICP/MS)
- Inductively Coupled Plasma/Optical Emission Spectroscopy (ICP/OES)
- Optical Emission Spectroscopy (OES)

Prior to use, verify that the above instrument(s) is in proper working order by following the instrument's *Performance Monitoring Protocol*. Instrumentation not listed above may be used, provided it is shown to be in proper working order prior to use. When the identity verification is completed, provide the applicable data and instrumental parameters to the CIM.

4.5.2 Purity Verification

Verification of the purity of a reference material will be performed after the identity verification and prior to quantitative use. It should be noted that, in addition to the techniques listed below, the use of established quantitative Positive Controls (from an approved standard operating procedure) are effective in verifying the purity of a reference material and allow for the verification of the reference material's stability after the identity verification.

A variety of techniques may be used to confirm the purity/concentration of the reference material. Only one technique is needed. Acceptable techniques for purity verification include:

- Gas Chromatography with applicable detector(s)
- Liquid Chromatography with applicable detector(s)
- Ultraviolet-Visible Spectroscopy (UV-Vis)

Prior to use, verify the above instrument(s) is in proper working order by following the instrument's *Performance Monitoring Protocol*. When the purity verification is completed, print the applicable data and instrumental parameters, have a second individual check the purity calculations (to include that individual's handwritten initials and date), and provide to the CIM.

4.5.2.1 Analytical Steps for Purity Verification

4.5.2.1.1 Gas Chromatography/Mass Spectrometry (GC/MS), Liquid Chromatography/Mass Spectrometry (LC/MS), and High Performance Liquid Chromatography (HPLC):

- Accurately dilute the new reference material to an appropriate concentration in an appropriate solvent (e.g., 100 µg/mL for GC/MS and 1-10 µg/mL for

LC/MS and HPLC).

- Accurately dilute a previously verified reference material of the same analyte, a previously calibrated deuterated analog of the same analyte, or a reference material of the same analyte from a different lot, to the same concentration in the same solvent used for the new reference material.

- Analyze the new diluted reference material solution with appropriate instrumental parameters. The analysis will be performed at least three times and the average area of the peak obtained.

- Following the same instrumental parameters, analyze the previously verified material solution, the deuterated material solution, or the solution of the reference material from a different lot.[1]

- Comparison of the average areas from the two reference materials allows for calculation of the concentration (and thus the purity) of the new reference material.

4.5.2.1.2 Ultraviolet-Visible Spectroscopy (UV-Vis)

- Accurately dilute the new reference material to 100 µg/mL (or other appropriate concentration) in an appropriate solvent.

- Analyze the new diluted reference material solution by UV-Vis. The UV-Vis analysis will be performed at least three times.

- Calculate the concentration of the diluted reference material solution using the Beer-Lambert law as follows:

$$A = abc$$

Where A = absorbance; a = absorptivity constant ($L \cdot g^{-1} \cdot cm^{-1}$); b = pathlength (usually 1 cm); and c = concentration of the sample (g/L)

- The specific absorbance constant of a 1% w/v solution of a chemical in a 1-cm cell (A_1) is available from *Clarke's Analysis of Drugs and Poisons* and other

[1] Optionally, a calibration curve of multiple points of the older reference material may be used in order to determine the purity of the new reference material.

references. The following equation can be used to calculate the absorptivity constant (a) from the specific absorbance constant (A_1):

$$a = \frac{A_1}{10}$$

- Substitution into the first equation (with b = 1 cm) results in:

$$c = A \times \frac{10}{A_1}$$

- Calculate the purity of the new reference material from the average concentration and the dilution factor used to prepare the solution.

4.6 Reference Material Verification Discrepancies

4.6.1 Discrepancies in Identity

Discrepancies in the structural identity of a reference material following qualitative testing will be discussed with the supplier and the material returned, if applicable. If the material is identified as something other than intended, the CIM must be notified. If the material is retained, the container must be labeled with information indicating the discrepancy. The supporting data will be provided to the CIM. Any previous data for that substance will be appropriately marked by the CIM.

4.6.2 Discrepancies in Purity

4.6.2.1 Discrepancies Within ±5% of the Listed Purity

If the purity verification of a reference material results in an average purity within ±5% of the listed purity, the listed purity will be used in preparing future calibrators or controls from this reference material.

4.6.2.2 Discrepancies Greater than ±5% of the Listed Purity

Discrepancies in the purity of a reference material greater than 5% of the listed purity will result in the assignment of an appropriate purity value (or concentration) to the material. This new purity (or concentration) will be recorded on the container and used in preparing future calibrators or controls from this reference material.

4.7 COAs and Other Records

COAs and other records associated with reference materials may be obtained through multiple sources. Records may be received in physical form along with the reference material. Alternatively, the records may be downloaded in electronic format from the supplier or manufacturer website. The applicable records and verification data will be maintained in the CU.

4.8 Records for Known Materials

When known materials are used in casework, sufficient information will be recorded in the case notes such that the nature of the known material is established. Examples of the type of information that may be necessary to record, if applicable, include:

- Product name
- Manufacturer name
- Universal Product Code (UPC)
- Lot number
- Expiration date
- Location purchased/acquired
- CU unique identifier for database samples

5 Calculations

Calculations used to determine the purity of reference materials are included in section 4.5.2.

6 Measurement Uncertainty

The *CU Procedures for Estimating Measurement Uncertainty* provides guidance for accounting for the uncertainty associated with the purity of reference materials.

7 Limitations

The limitations associated with this procedure are dependent on the instrumental techniques used to determine the identity and purity of reference materials. In general, the listed techniques will be sufficient for these determinations when performed as described.

8 Safety

Take precautions for the handling of all chemicals. Refer to appropriate SDS for safe handling practices. Refer to the *FBI Laboratory Safety Manual* for guidance.

9 References

CU Procedures for Measurement Traceability

FBI Laboratory Operations Manual

CU Procedures for Estimating Measurement Uncertainty

FBI Laboratory Safety Manual

Clarke's Analysis of Drugs and Poisons, Pharmaceutical Press (multiple editions, also available online)

The Merck Index, RSC Publishing (multiple editions, also available online)

Instrumental Data for Drug Analysis, T. Mills, J.C. Roberson, C.C. Matchett, M.J. Simon, M.D. Burns, and R.J. Ollis, Jr., 3rd ed., Volumes 1-6, CRC Press: Boca Raton, Florida, 2006.

Rev. #	Issue Date	History
7	02/09/18	Changed title to include 'known materials'. Added existence of metal reference materials that are not CRMs but don't require verification in section 1 and section 4.5, also added information on 'known materials' throughout. Removed previous sections 4 and 5 (Calibration, Sampling) since not required. Added last paragraph to new section 3. Edited section 4.1 for clarity and removed "or designee", also changed "document" to "record". Removed first sentence from section 4.2. Added FD-455 and dual-person entry information; removed "or designee" and replaced with "Unit Chief"; clarified that the keys are assigned (not that "access to controlled substances is restricted" in general); and pluralized "container" within section 4.2.1. Created fourth bullet in section 4.4 for clarity. Used CU abbreviation in section 4.7. Added section 4.8. Edited section 5 to clarify that not "all" calculations are necessarily accounted for.
8	09/13/19	Removed "Subunit" from "Metallurgy" in first paragraph, and edited the details of Metallurgy reference materials for clarity. Changed "shall" to "will" in section 3 (second paragraph, first sentence). Revised section 4.1 to include the CU database and purchase credit card holder responsibility. Revised second paragraph of section 4.1 for clarity. Removed the use of the CU Controlled Substance Log from section 4.2.1 and remaining sections including previous Appendix A (weight information will be recorded in CU database). Removed several storage requirements from section 4.2.1 since the requirements are no longer necessary. Added last bullet to section 4.4. Removed the need to print applicable information to be provided to the CIM in section 4.5.1. Changed "MSDS" to "SDS" in section 8.

Approval

Redacted - Signatures on File

General Chemistry
Technical Leader: Date: 09/11/2019

Acting Toxicology
Technical Leader: Date: 09/11/2019

Metallurgy
Technical Leader: Date: 09/11/2019

Paints and Polymers
Technical Leader: Date: 09/11/2019

Chemistry Unit Chief: Date: 09/11/2019

QA Approval

Quality Manager: Date: 09/11/2019

Chemistry Unit
Procedures for Verification of Reagents

1 Purpose

A reagent is a substance used because of its known chemical or biological activity. These procedures describe the verification and labeling of these items when used for casework in the Chemistry Unit (CU).

2 Scope

This document applies to CU personnel that use reagents for casework in all disciplines/categories of testing with the exception of Fire Debris.

3 Equipment/Materials/Reagents

The appropriate equipment and materials used to verify a reagent will depend upon the nature of the substance. In most instances the verifications will be performed following an established CU standard operating procedure (SOP), and the equipment, materials, and reagents required will be listed within the applicable SOP(s).

4 Procedures

4.1 Procedures for Verification of Reagent Reliability

The reliability of a reagent will be verified prior to, or in concurrence with casework. This may be done in any of the following ways:

- When available, follow the reagent verification instructions given in the SOP for the particular analysis in which the reagent is used.
- Perform the analysis using suitable standards, controls, and/or blanks and evaluate the outcome.
- Measurement of a chemical property (e.g., pH).
- Apply to an item and evaluate a physical property (e.g., contrast of microstructural phases).

Any applicable reagent verification data acquired will be kept in an appropriate location, such as

within a reagent logbook, an instrumentation binder, data archive, and/or case notes.

4.2 Labeling of Reagent Containers

4.2.1 Unit-Prepared Reagents

Reagents prepared in the CU will be recorded using the CU *Reagent Preparation Log* (Appendix A). Additionally, the following will be recorded on the container of a reagent prepared in the CU:

- Reagent name (using common name or SDS name)
- Lot number[1]
- Expiration date, if applicable

4.2.2 Purchased Reagents

It is good laboratory practice to record the date received, date opened, and opener's initials on the reagent container, however this is not a requirement.

4.3 Reagent Preparation Records

For each prepared, stored reagent, the following information will be recorded in a reagent log book maintained by each group (see Appendix A):

- Date of preparation
- Preparer
- Lot number
- Components used to make the reagent and their source and lot information
- Person verifying (may be the same as the preparer), date of verification, and result of verification
- Expiration date, if applicable as determined by individual SOP

4.4 Use of Reagents Beyond Their Listed Expiration Date

Reagents may be used past their expiration dates provided that appropriate steps are taken with every use to demonstrate reliability (see section 4.1). The expiration date will not be altered or

[1] Lot numbers for reagents prepared in-house will contain the initials of the person preparing the reagent and the date of preparation. If multiple reagents are made on the same day by the same person, a letter will be added to the end of the lot number to ensure a unique link to the appropriate reagent.

removed.

4.5 Recording of Reagent Lot Number

The lot number of any reagent(s) used to examine evidence will be recorded in the case notes.

5 Safety

Take standard precautions for the handling of all chemicals. Refer to appropriate Safety Data Sheets (SDS) for safe handling practices. Refer to the *FBI Laboratory Safety Manual* for guidance.

6 References

FBI Laboratory Safety Manual

Rev. #	Issue Date	History
5	02/09/18	Edited section 1 for brevity. Revised scope to include personnel. Reworded last sentence in Section 3 for clarity. Removed previous sections 4 and 5 (calibration and sampling). Section 4.1- simplified third bullet, added fourth bullet, and expanded on storage locations for verification data. Changed "document" to "record" in section 4.2.1. Edited section 4.2 for clarity. Changed "MSDS" to "SDS" throughout. Edited section 4.4 to not allow alteration/removal of an expiration date and relied on reference to section 4.1. Added section 4.5. Revised Appendix A.
6	09/13/19	Removed "mixed solvents" and "buffers" from section 1. Changed "subunit" to "group" in section 4.3. Simplified footnote. Removed five references from section 6 (outdated and not needed).

Approval

Redacted - Signatures on File

General Chemistry
Technical Leader: Date: 09/11/2019

Acting Toxicology
Technical Leader: Date: 09/11/2019

Metallurgy
Technical Leader: Date: 09/11/2019

Paints and Polymers
Technical Leader: Date: 09/11/2019

Chemistry Unit Chief: Date: 09/11/2019

QA Approval

Quality Manager: Date: 09/11/2019

Redacted - File on File

Chemistry Unit
Document Control Procedures

1 Purpose

These procedures detail the methods used in the Chemistry Unit (CU) to comply with the *FBI Laboratory Quality Assurance Manual* and *FBI Laboratory Practices for Document Control.*

2 Scope

These procedures apply to personnel that prepare and use CU controlled documents. These include Standard Operating Procedures (SOPs), Instrument Operation and Systems Support (IOSS) Protocols, *CU Quality Assurance and Operations Manual* documents, *CU Training Manual* documents, and equipment manuals/externally produced quality documents that CU personnel are required to follow for specific procedures or instructions.

3 Document Distribution and Control

3.1 SOPs

SOPs, including forms, posted on BUNET and LABNET will be the official (controlled) versions. CU personnel will also have access to controlled copies of SOPs on the CU local area network (CHEMNET).

3.2 IOSS Protocols

IOSS Protocols posted on BUNET and LABNET will be the official (controlled) versions. CU personnel will also have access to controlled copies of IOSS Protocols on CHEMNET.

3.3 *CU Quality Assurance and Operations Manual* Documents

CU Quality Assurance and Operations Manual documents, including forms, posted on BUNET and LABNET will be the official (controlled) versions. CU personnel will also have access to controlled copies of *CU Quality Assurance and Operations Manual* documents, but not associated forms, on CHEMNET.

3.4 Instrumentation Manuals and Externally Produced Quality Documents

Instrumentation manuals and other externally produced quality documents (to include electronic versions of these manuals/documents) that CU personnel are required to follow for specific procedures or instructions will be controlled. This will be accomplished by labeling each applicable manual/document as "controlled" on at least the first page or cover of the manual/document or directly on the electronic storage device containing the manual/document. Additionally, a review signature and an approval signature are required, along with the dates of the signatures. For electronic manuals/documents, these signatures and dates are recorded within the *CU Electronic Instrument Manuals Under Document Control Review and Approval Document*, which is stored in the CU Chief's office. Each manual and document will be assigned to at least one individual in the CU. A list of these manuals and documents will be maintained in the CU Chief's office and will serve as the master list of these controlled documents. Archiving of these manuals and documents will follow the *FBI Laboratory Practices for Document Control*.

4 Working Electronic Copies of CU Generated Quality System Documents

Working electronic copies of the documents listed below are maintained by the indicated personnel. The electronic files are to be used for the preparation of document revisions and are secured to prevent unauthorized editing.

4.1 SOPs

Electronic files are maintained by the appropriate Technical Leader and the CU Chief.

4.2 IOSS Protocols and IOSS-Specific *CU Training Manual* Documents

Electronic files are maintained by the Instrument Manager and the CU Chief.

4.3 *CU Quality Assurance and Operations Manual* Documents

Electronic files are maintained by the CU Quality Assurance Program Manager and the CU Chief.

4.4 General *CU Training Manual* Documents

Electronic files are maintained by the CU Training Program Manager and the CU Chief.

4.5 Category of Testing/Discipline-Specific *CU Training Manual* Documents

Electronic files are maintained by the appropriate Technical Leader and the CU Chief.

5 Annual Review of CU Controlled Documents

An annual review of the documents listed below will be ensured by the indicated personnel. The annual review will be recorded in an Annual Review Memo issued to the CU Chief by the end of each calendar year. Personnel responsible for more than one area may combine those areas into one Annual Review Memo. The Annual Review Memo will contain the following information in addition to the annual review information required by the LOM (see *FBI Laboratory Practices for Document Control*):

- Any planned revisions, along with a timeline for submission of the revised document(s)
- For any document that does not require revision, a statement indicating such

5.1 SOPs

Each Technical Leader will issue an Annual Review Memo for his/her applicable SOPs, IOSS Protocols, and *CU Training Manual* documents.

5.2 IOSS Protocols

The Instrument Manager will issue an Annual Review Memo for the IOSS Protocols and his/her applicable *CU Training Manual* documents.

5.3 *CU Quality Assurance and Operations Manual* Documents

The CU Quality Assurance Program Manager will issue an Annual Review Memo for the *CU Quality Assurance and Operations Manual* Documents.

5.4 General *CU Training Manual* Documents

The CU Training Program Manager will issue an Annual Review Memo for the general *CU Training Manual* Documents.

5.5 Instrumentation Manuals and Externally Produced Quality Documents

Each Technical Leader and the Instrument Manager will issue an Annual Review Memo for his/her applicable instrumentation manuals and/or externally produced quality documents.

6 Records

The following records will be generated and/or permanently retained in CU as a result of these practices:

- *Document Review Form* [retained in the Forensic Analysis Support Unit (FASU)]
- *CU Electronic Instrument Manuals Under Document Control Review and Approval Document*
- Master list of controlled instrumentation manuals and externally produced quality documents
- Annual Review Memos to the CU Chief

Rev. #	Issue Date	History
8	07/12/18	Added new material covering annual review of CU controlled documents (see section 5 and last bullet in section 6). Revised the first paragraph and the format of section 4 to be consistent with section 5.
9	09/13/19	Added IOSS Protocols to section 2 and throughout to differentiate from "SOPs". Removed "CU" from "CU personnel" in sections 4 and 5 (IOSS Manager is no longer assigned to CU due to reorganization). Changed title of sections 4.2 and 5.2 for consistency. Added IOSS Protocols to section 5.1. Changed "Training Modules" to "*CU Training Manual* document" throughout. Removed previous section 5.5 as content integrated into sections 5.1 and 5.2.

Approval

Redacted - Signatures on File

General Chemistry
Technical Leader: Date: 09/11/2019

Acting Toxicology
Technical Leader: Date: 09/11/2019

Metallurgy
Technical Leader: Date: 09/11/2019

Paints and Polymers
Technical Leader: Date: 09/11/2019

Chemistry Unit Chief: Date: 09/11/2019

QA Approval

Quality Manager: Date: 09/11/2019

Chemistry Unit
Validation of Analytical Procedures

1 Purpose

This document supplements the *FBI Laboratory Practices for Developing Methods and Validating Technical Procedures, FBI Laboratory Practices for Validating Chemical Procedures,* and the *FBI Laboratory Quality Assurance Manual* for the validation of new analytical procedures in the Chemistry Unit (CU).

2 Scope

This document applies to personnel that validate new analytical casework procedures in CU disciplines/categories of testing with the exception of Fire Debris. Validation starts after a method is acquired or developed. If a method needs to be developed in CU (including the modification of an acquired method), the method development will be a planned activity. The method development plan must be recorded, approved by the applicable Technical Leader, and any changes to the plan will be communicated to all personnel involved in the method development.

The validation of an analytical procedure is referred to as a validation study in the CU. The performance characteristics that are evaluated during a validation study will be based on the scope of the analytical procedure. The validation study must be completed, reviewed, and approved prior to the procedure's first use in casework, except as noted within this procedure.

3 Responsibilities

3.1 The Individual/Group performing the method development or validation study will:

- Develop, record, and ensure approval of a method development plan utilizing the CU *Method Development Plan* form (Appendix A). Retain the approved CU *Method Development Plan* form.
- Record and/or reference any other technical work relied upon to support the usage of a novel methodology or process.
- Record and retain the results of the method development.
- Develop, record, and ensure approval of a validation plan utilizing the applicable CU *Validation Plan* form (Appendices B-D). Retain the approved CU *Validation Plan* form.
- Record and retain the results of the validation study. The supporting records may include validation data, instrument optimization charts, calculations, relevant literature references, etc.

- Ensure technical review and approval of the validation study utilizing the *Validation of Chemical Procedures Review Form* (7-267) or the CU *Validation Plan and Review- Physical Properties Only* form (Appendix D), as applicable.
- Complete a *Validation Summary* form (Appendix E).
- Maintain all supporting records related to method development and validation studies conducted within the CU within binders, in electronic format, and/or within case notes, as appropriate. Method development and validation study binders will be stored within the CU's file cabinets and/or bookshelves.

3.2 The applicable Technical Reviewer(s) will:

- Review and approve validation records. Record these reviews on the 7-267 or CU *Validation Plan and Review- Physical Properties Only* form (when applicable).

3.3 The applicable Technical Leader will:

- Review and approve method development plans by signing the CU *Method Development Plan* form. If the Technical Leader is the "Lead Scientist" for the method development, then another CU member qualified in the discipline/category of testing (if available) will review and approve the method development plan.
- Review and approve validation plans by signing the applicable CU *Validation Plan* form. If the Technical Leader is the "Lead Scientist" for the study, then another CU member qualified in the discipline/category of testing (if available) will review and approve the validation plan.
- Approve validation studies by signing the "Technical Leader Approval" line on the 7-267 (when applicable) or the CU *Validation Plan and Review- Physical Properties Only* form. If the Technical Leader is the "Lead Scientist" for the study, then another CU member qualified in the category of testing (if available) will sign the CU *Validation Plan and Review- Physical Properties Only* form.
- Approve validation summaries by signing the CU *Validation Summary* form.

3.4 The CU Chief will:

- Approve validation plans by signing the CU *Validation Plan* form.
- Approve validation studies by signing the 7-267 or the CU *Validation Plan and Review- Physical Properties Only* form (when applicable).
- Approve validation summaries by signing the CU *Validation Summary* form.

4 Procedures

4.1 Validation Studies

Validation studies of chemical procedures in the CU will be performed following the requirements outlined in the *FBI Laboratory Practices for Validating Chemical Procedures.* These requirements may be adjusted based on the scope of the procedure and professional judgment (e.g., safety considerations, differences in sample matrices, availability of reference

materials).

Validation studies of applicable, non-chemical procedures (i.e., physical property measurements) in the CU will be limited to the characteristic listed in section 4.1.1 of this document.

Validation studies for casework involving the analysis of unknowns will be conducted as detailed in section 4.1.2 of this document.

4.1.1 Performance Characteristic for Measurement of a Physical Property

4.1.1.1 Accuracy

Accuracy is the closeness of an analytical result to its true value and is affected by systematic error (bias) and random error (precision). The accuracy of a physical property measurement can be determined by comparison of that measurement result with the true value. At a minimum, ten measurement replicates of a reference material with a known physical property value are made. The accuracy is calculated as the percent difference of the average measured value from the known value. In most instances, the preferred accuracy is ±15% or less, but larger values may be unavoidable and are acceptable if accompanied by proper justification.

4.1.2 Performance Characteristics for the Analysis of Unknown Component(s)

Due to the nature of unknown component analysis, the validation may be conducted either at the time of analysis or immediately following. Generally, the only performance characteristic that needs to be validated is interferences.[1]

4.2 Record, Review, and Maintain Validation Study Results

Upon completion of the validation study, the CU Chief or appropriate Technical Leader will assign appropriate personnel as technical reviewer(s) of the validation study results. If the CU Chief is qualified to do so, he/she may perform the technical review. The technical review must take place before the procedure is placed into use.[2] The technical reviewer(s) will complete the 7-267 or the CU *Validation Plan and Review- Physical Properties Only* form, whichever is applicable. After the technical review is complete, the CU Chief and appropriate Technical Leader (as applicable) will review the validation records and record their approval on the 7-267 or the CU *Validation Plan and Review- Physical Properties Only* form. The completed review form will be maintained with the validation study data. Once a new analytical procedure has final approval by the CU Chief, and before it is used in casework, the procedure will be formally

[1] In this scenario, the requirements of completing a validation plan and 7-267 will be waived.

[2] The exception to this rule will be when validating the analysis of an unknown component. In these cases, the 7-267 will not be required and the review of the validation will occur as part of the *Laboratory Report* technical review.

written and reviewed following the appropriate Laboratory Division and CU practices.

In extreme situations (e.g., court mandates) when a validated procedure must be used prior to being formally written and through all reviews, it is permissible to use the validated procedure for casework provided that the same steps for sample preparation and instrumental parameters used during the validation are also used for the analysis and there is a clear, written record of the steps that were taken to generate the results. In these instances, at a minimum, the validation data will be technically reviewed by another qualified examiner in the category of testing prior to using the validated procedure for casework. This will be treated as a minor or major deviation, as appropriate, according to the *FBI Laboratory Practices for Authorizing Deviations*.

Validation study records will be maintained within the CU's validation file cabinets, bookshelves, in electronic format, or, with respect to case-specific validation, in the related case notes.

When a validation study has been performed for what is most likely to be a one-time analysis, a validated procedure can be applied in casework without the issuance of an official standard operating procedure. In these instances, the following criteria will be met:

- A validation plan will be created and technically reviewed using the appropriate CU *Validation Plan* form, and approved prior to commencing validation.
- Step-by-step instructions for the analysis and a summary of the validation performed will be prepared and retained with the validation records.
- The validation records will be technically reviewed and approved by the CU Chief and appropriate Technical Leader, if applicable. This will be recorded on the 7-267 or on the CU *Validation Plan and Review- Physical Properties Only* form, whichever is applicable.
- A copy of the applicable review form and a copy of the step-by-step instructions will be retained in the case notes for the affected case.
- The validation records will be stored electronically and/or in a central location to include the CU's validation file cabinets or bookshelves.
- If and when the procedure is performed again, a standard operating procedure will be written. Required reviews and approvals will be obtained before issuance of the new procedure.

4.3 Validation Summary

A *Validation Summary* form (Appendix E) will be completed for each CU validation study that results in a new CU standard operating procedure. The individual that led the validation study will complete the form and provide it to the applicable Technical Leader. The summary will briefly describe the performance characteristics that were evaluated to include the values that were obtained for the performance characteristics, if applicable. Other details may be included in the summary. An abstract for a scientific article is a basic model that may be considered when

composing the summary.

5 Competency Testing on Newly Validated Analytical Procedures

Caseworking personnel must successfully complete a competency test on a newly validated analytical procedure prior to applying the procedure to casework. This test will demonstrate that applicable personnel can accurately perform the procedure. For personnel that were involved in the validation process, the CU Chief and appropriate Technical Leader may approve the validation work to serve as demonstration of competency. The successful completion of a competency test, or the approval to use validation work as a substitute for a competency test, will be recorded in the employee's Training and Qualification Records binder.

6 Minor Deviations to Previously Validated Procedures

Minor deviations to standard operating procedures in the CU will be considered for approval by the requestor's Technical Leader, and approved prior to the minor deviation being employed. If the requestor is a Technical Leader, the minor deviation request will be considered for approval by the CU Chief. If necessary, the CU Chief will consult with an examiner that is qualified in the procedure and both the CU Chief and the consulted examiner will record their approval of the minor deviation.

6.1 Minor Deviation Records

To maintain consistency when other CU personnel are faced with the same or similar analyses, all minor deviations to standard operating procedures will be recorded by the applicable Technical Leader in a centralized location. The format of the records is left to the discretion of the Technical Leader. At a minimum, the records will include the following:

- FBI Laboratory number associated with the minor deviation
- Date of the minor deviation
- Personnel that performed the minor deviation
- Personnel that approved the minor deviation
- Title of the document (or unique identifier), issue date and/or revision number, and the specific requirement(s) from which a minor deviation is sought
- A statement of the specific deviation
- This will allow for review of the minor deviation if the original records reside in a specific case file

7 References

FBI Laboratory Practices for Validating Chemical Procedures, FBI Laboratory Operations Manual.

FBI Laboratory Practices for Developing Methods and Validating Technical Procedures

LeBeau, M. et al. "Validation Guidelines for Laboratories Performing Forensic Analysis of Chemical Terrorism", *Forensic Science Communications,* 7(2), April 2005.

Peters, F.T. "Bioanalytical Method Validation and its Implications in Forensic and Clinical Toxicology - A Review", *Accred. Qual. Assur.* 7, 2002, 441-449.

Peters, F.T., Drummer, O.H., and Musshoff, F. "Validation of New Methods", *Forensic Science International,* 165(2-3), 2007, 216-224.

Rev. #	Issue Date	History
9	02/09/18	Edited section 1 to reflect changes to titles of LOM documents. Edited section 2 to include personnel and added "casework" to first sentence in section 2 (to differentiate from other procedures, such as instrumentation). Changed "and" to "and/or" in first two sentences in section 2; and in first sentence in fourth bullet of section 3.1. Changed "document" and related to "record" and related throughout. Changed "Examiner" to "examiner" throughout. Added reference to *Validation Summary* form in sections 3.1, 3.3, and 3.4; added Appendix D (*Validation Summary* form). Changed "Subunit Manager" to "Technical Leader" throughout. Added validation plan to 1st footnote. Italicized "Laboratory Report" in 2nd footnote. Added section 4.3. Edited last sentence of section 6 for clarity. Minor edits made to section 6.1. Revised reference to account for merger of Level 1 documents.
10	09/13/19	Separated 'method development', 'develop', etc. from 'validation' throughout to account for the requirement to have method development activities approved and recorded; and to clarify that these are separate activities in CU. Removed bullet from section 4.1.1. Revised section 6.1 to align with Level 1 documents. Added Appendix A- CU *Method Development Plan*.

Approval

Redacted - Signatures on File

General Chemistry
Technical Leader: Date: 09/11/2019

Acting Toxicology
Technical Leader: Date: 09/11/2019

Metallurgy
Technical Leader: Date: 09/11/2019

Paints and Polymers
Technical Leader: Date: 09/11/2019

Chemistry Unit Chief: Date: 09/11/2019

QA Approval

Quality Manager: Date: 09/11/2019

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Chemistry Unit
Procedures for Training and Continuing Education

1 Purpose

Training and continuing education is of the utmost importance for developing and maintaining a qualified and competent technical staff. This document details the procedures for training and continuing education within the FBI Laboratory's Chemistry Unit (CU). Forensic examiner trainees, as well as their training committee members, will also refer to the *FBI Laboratory Practices for the Forensic Examiner Training Program* for appropriate requirements.

2 Scope

This document applies to training and continuing education for all CU employees in all disciplines/categories of testing with the exception of Fire Debris.

3 Records

CU personnel training records will be maintained within binders in the CU Chief's office, electronically by the CU Training Program Manager, and/or in Virtual Academy.

Trainees will maintain a daily training log and must fulfill the training requirements as outlined in his or her personal training plan. An examiner trainee's training log must record how many hours were spent on specific activities and the kind of training that took place, either direct instruction (with an accounting for who gave the instruction) or self-study. For a non-examiner trainee, a monthly memo will be provided to the CU Chief by the trainee's mentor or the CU Training Program Manager. The memo will summarize the trainee's development and progress.

4 Training and Continuing Education

4.1 Training New Technical Personnel

New CU technical personnel will complete the CU training program that includes general and technical instruction. Each trainee will be assigned a training committee to include a mentor that will serve as the trainee's primary instructor. The training committee, along with the CU Training Program Manager, CU Chief, and other assigned CU personnel will work with the trainee to develop a customized written training plan that will outline qualification requirements. When hiring experienced technical personnel, the CU Chief and applicable Technical Leader are responsible for assessing the Trainee's previous training and ensuring it is adequate and recorded. Modification(s) to the CU training program may be appropriate and will be recorded

in the training plan. If the modification(s) results in fewer than the minimum requirements indicated in the *Practices for the Forensic Examiner Training Program, Practices for Oral Board Exercises*, or *Practices for Moot Court and Admissibility Hearing Exercises* then the modification(s) require approval by the Forensic Examiner Training Program Manager (FETPM) prior to implementation.

Each trainee will be assigned specific modules to complete within the *CU Training Manual*. The version posted on BUNET and LABNET will be the official (controlled) version of the manual. Trainees may print copies of the *CU Training Manual* from BUNET or LABNET for use as part of his/her training materials. In order to complete each assigned module, the trainee will have to successfully pass a test (oral, written, or both) in that subject area. Further, trainees conducting casework will receive training samples, as well as competency tests, and will be required to demonstrate competency by analyzing these samples correctly.

4.2 New Subject Area Training of Experienced Technical Personnel

As the needs of the CU change, it is sometimes necessary for experienced technical personnel to undergo training in a new discipline and/or category of testing. When this occurs, the training program will follow the basic design of the CU training program, however it may be modified to avoid duplication of training topics.

4.3 Continuing Education

CU technical personnel will complete a minimum of 15 hours of relevant continuing education each fiscal year, while non-technical personnel (e.g., MAPA) will complete a minimum of 8 hours each fiscal year. The nature of the continuing education will be recorded within Virtual Academy. For continuing education activities that are not automatically accounted for within Virtual Academy, the Self-Reported Training feature will be used to create a record within Virtual Academy.

Continuing education training topics and courses in the CU are chosen by the employee and his/her supervisor. Acceptable topics of training must directly relate to the employee's current job requirements and should focus on maintaining technical skills and expertise. For example, these topics may include instrumental courses, relevant workshops at scientific meetings, literature review, or management courses. Approval of a continuing education topic is granted by an employee's supervisor and/or the CU Chief.

4.4 Remedial Training

At times, it may be necessary to provide remedial training for experienced technical personnel (e.g., failed proficiency test, improper use of a validated protocol). In these instances the *FBI Laboratory Practices for Addressing a Nonconformity* will be followed and a remedial training plan will be developed by the CU Chief and applicable Technical Leader for the affected employee.

Rev. #	Issue Date	History
6	02/09/18	Changed “document” (and related) to “record” (and related); changed “Examiner” to “examiner”; and changed “Subunit Manager” to “Technical Leader” throughout. Changed “or designee” to “or the CU Training Program Manager” in section 3. Edited section 4.1 to include FETPM approval of certain modifications. Edited last paragraph of section 4.1 for clarity. Added “literature review” to second paragraph of section 4.3. Created section 4.3.1 and edited content for clarity (was previously contained in section 4.3).
7	09/13/19	Removed training course evaluation requirement (previous section 4.3.1 and Appendix A form).

Approval

Redacted - Signatures on File

General Chemistry
Technical Leader: Date: 09/11/2019

Acting Toxicology
Technical Leader: Date: 09/11/2019

Metallurgy
Technical Leader: Date: 09/11/2019

Paints and Polymers
Technical Leader: Date: 09/11/2019

Chemistry Unit Chief: Date: 09/11/2019

QA Approval

Quality Manager: Date: 09/11/2019

Chemistry Unit
Procedures for Estimating Measurement Uncertainty

1 Purpose

No measurement is exactly known. Measurement uncertainty is the variability associated with a quantitative measurement result based on the information known about the measurement method. This document describes the Chemistry Unit's (CU) approach to estimating measurement uncertainty. The approach is based on a simplified version of the "Guide to the Expression of Uncertainty in Measurement" or the "GUM"; a widely-accepted method for evaluating, estimating, and expressing measurement uncertainty, as well as the National Institute of Standards and Technology (NIST) 8-Step Process.

2 Scope

This document applies to CU personnel recording and/or reporting measurement results that require an estimation of measurement uncertainty. Measurement uncertainty will be estimated for all reported quantitative results.

Additionally, measurement uncertainty will be estimated and reported for the following conditions:

- The measurement uncertainty is relevant to the validity or interpretation of the examination results.
- The measurement uncertainty is required by the contributor.
- The measurement uncertainty affects compliance to a specification limit.

3 Records

The following supporting records related to the estimation of measurement uncertainty will be maintained. This information may be recorded in multiple locations to include: standard operating procedures, validation binders, measurement uncertainty records (to include electronic files, e.g., Excel® spreadsheets), and case files.

- Statement defining the measurand (i.e., the quantity intended to be measured)
- Statement of how traceability is established for the measurement
- The equipment [e.g., measuring device(s) or instrument(s)] used
- All uncertainty components considered

- All uncertainty components of *significance* (see section 4.2) and how they were evaluated
- Data used to estimate repeatability, intermediate precision, and/or reproducibility
- All calculations performed
- The combined standard uncertainty, the coverage factor (k), the confidence level (also known as the coverage probability) and the resulting expanded uncertainty
- The schedule to review and/or recalculate the measurement uncertainty

4 Estimating Measurement Uncertainty

The eight steps listed below are used to estimate measurement uncertainty in the CU:

- Step 1: Specify the measurement process
- Step 2: Identify uncertainty components
- Step 3: Quantify uncertainty components
- Step 4: Convert quantities to standard uncertainties
- Step 5: Calculate combined standard uncertainty
- Step 6: Expand the combined standard uncertainty by coverage factor (k)
- Step 7: Evaluate the expanded uncertainty
- Step 8: Report the uncertainty

The CU utilizes uncertainty budgets for performing estimation of measurement uncertainty calculations. An example spreadsheet is shown in Figure 1.

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4.1 Step 1: Specify the Measurement Process

In the first step, the measurand is defined. The measurand is the quantity intended to be measured. It is important to be as specific as possible when defining the measurand. The measurand will likely be determined by a combination of measurement processes. If necessary, include a reference to a specific standard operating procedure, instrument, etc., in the statement defining the measurand to distinguish one measurement process from another.

4.2 Step 2: Identify Uncertainty Components

Possible uncertainty components associated with the measurement process should be assembled into a reasonably comprehensive list. This list must include all uncertainty components considered, and which uncertainty components were deemed to be *significant*. An uncertainty component is considered *significant* if a change in the uncertainty component corresponds to a

change in the significant figures of the stated value or uncertainty of the measurement result. Several uncertainty components that may be considered in this process are provided below.

The specific measuring device or instrument used for a reported test result must be evaluated in the estimation of measurement uncertainty for the associated test method.

- Sampling (homogeneity, physical state, environment, etc.)
- Sample preparation (homogenizing, dissolving, extracting, diluting, concentrating, derivatizing, etc.)
- Reference materials (purity, ability to matrix match, etc.)
- Uncertainty of a calibration (pipettes, balances, etc.)
- Calibration curves (uncertainty of calibrators, matrix matching of calibrators, etc.)
- Analysis (systematic errors, random errors, environment, matrix interferences, run-to-run precision, etc.)

4.2.1 Reconciliation of Uncertainty Components

Reconciliation simplifies the uncertainty budget. In this step, a review is conducted to determine whether a listed uncertainty component is adequately accounted for by existing data (usually repeatability data) or small experiments are planned to account for the uncertainty component. The basis for this step lies in the fundamental assumption that if an uncertainty component is representatively varied during the course of a series of observations, then the uncertainty associated with that component is adequately accounted for in the repeatability of those observations. Of course, it is important that those uncertainty components that are reconciled in this step are truly represented through the existing data or planned experiments.

4.3 Step 3: Quantify Uncertainty Components

Once the uncertainty components have been identified and reconciled, the standard deviation of each will be determined. The approach to calculating the standard deviation is dependent on whether the uncertainty component is classified as a *Type A* or *Type B*.

4.3.1 *Type A* Uncertainty

Type A uncertainty is evaluated by the statistical analysis of data from a series of measurements, assuming a normal distribution. The CU relies on the use of "historical" data (e.g., method validation data, positive control data) to establish a historical standard deviation for the measurement process. The historical standard deviation is the value assigned to the *Type A* uncertainty associated with the measurement process and the equation for calculating the historical standard deviation (s_{hist}) is shown below. This standard deviation may be referred to interchangeably in the CU by a variety of terms including: historical standard deviation, sample

standard deviation, method standard deviation, process standard deviation, and standard deviation. Additionally, the use of the symbol, σ (traditionally used to indicate the population standard deviation) in place of the symbol, s, when referring to a sample standard deviation in the CU is acceptable as long as the associated calculations are clearly defined.

$$s_{hist} = \sqrt{\frac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}} \ ,$$

where $\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$ (i.e., $\bar{x}$ is the average measurement result),

and $n =$ the number of measurements

There may be instances where the standard deviation for a measurement process is calculated to be extremely small or even zero due to the standard deviation being less than the resolution of the measuring device. In these instances, the estimated standard deviation (s_p) will be calculated from the below equation, where d = the measuring device resolution. The estimated standard deviation will be compared to the observed standard deviation and the larger value will be used.

$$s_p = \frac{d}{\sqrt{3}}$$

4.3.1.1 Measurement Assurance and Updating the Historical Standard Deviation

At least one positive control sample is analyzed with each measurement process. A range of acceptable values for positive control samples is defined in the associated CU standard operating procedures. If a value that does not fall within the acceptable range is observed, then the result will be investigated. If the value cannot be explained (e.g., human error, instrument malfunction) then an appropriate statistical analysis will be performed to determine if the value is an outlier. An outlier value will be rejected and not used to calculate the updated standard deviation. Otherwise the value will be included in the updated standard deviation calculation.

The schedule to update the repeatability component (i.e., standard deviation) used in uncertainty calculations will be defined within standard operating procedures.

4.3.1.2 Adjustments to the Historical Standard Deviation when Reporting the Average of Multiple Measurements of a Case Specimen (Standard Deviation of the Mean)

It is common for multiple measurements of a case specimen to be made and the average of the multiple measurements to be reported. These repeat measurements provide more information and more confidence in the reported result. In these instances, the standard deviation of the mean (s_{mean}) will be calculated as follows, where s_{hist}= the historical standard deviation, and n

= the number of measurements used to calculate the average value of the case specimen:

$$s_{mean} = \frac{s_{hist}}{\sqrt{n}}$$

The standard deviation of the mean is then used as the *Type A* uncertainty value.

As an example, if a historical standard deviation for a procedure was equal to 4.3_8% and a case specimen measurement result was based on an average of 5 measurements, then the standard deviation of the mean would be calculated as [$(4.3_8\%) / \sqrt{5}$] = 1.9_6%. This value of 1.9_6% would then be used as the *Type A* uncertainty value in the estimation of measurement uncertainty calculations (note- in this example three significant figures are carried forward as indicated by the subscript in the hundredths place, with the intention of rounding up to two significant figures at the conclusion of the uncertainty calculations).

4.3.2 *Type B* Uncertainty

Type B uncertainty is evaluated by means other than the statistical analysis of data from a series of observations. No single approach is applicable for evaluating and quantifying these uncertainty components. Examples of *Type B* uncertainty components include uncertainty of a calibration (i.e., external calibration services), uncertainty of a reference material, and uncertainty of volumetric glassware.

Some *Type B* uncertainty values can be derived from sources of information that are readily available in the CU. These sources include:

- Calibration certificates of reference materials, instrumentation, and equipment
- Manufacturer's specifications for volumetric glassware, instrumentation, and equipment
- Reference data from handbooks

When information sources such as those listed above are not available for deriving *Type B* uncertainty values, but the upper and lower limits of the instrument or device are known, then the uncertainty value will be estimated using the Rectangular Distribution or Triangular Distribution approaches described below. When in doubt, use the Rectangular Distribution approach as it is the more conservative approach.

4.3.2.1 *Type B* Uncertainty- Rectangular Distribution

A Rectangular Distribution approach can be used to estimate a *Type B* uncertainty component if the following criteria are met: the upper and lower limits of the instrument or device are known,

the probability that a value lies outside of these limits is zero, and one value is just as likely as another value between the limits (equal probability). For a Rectangular Distribution, the upper limit = +a, the lower limit = -a, and the possible range of values = (+a) – (-a) = 2a. The calculation to estimate the equivalent of one standard deviation is defined as:

$$s = \frac{a}{\sqrt{3}}$$

For example, if a 100 mL volumetric flask has a tolerance of ±0.2 mL, then the upper limit = +0.2 mL, the lower limit = -0.2 mL, and the range of the outer limits = 0.4 mL. The estimated standard deviation is calculated as:

$$s = \frac{0.2\ mL}{\sqrt{3}} = 0.1_2\ mL$$

4.3.2.2 Type B Uncertainty- Triangular Distribution

A Triangular Distribution approach can be used to estimate a *Type B* uncertainty component if the following criteria are met: the upper and lower limits of the instrument or device are known and a value near the center is more likely than one at the upper or lower limit. For a Triangular Distribution, the upper limit is still equal to +a, and the lower limit is still equal to -a. The calculation to estimate the equivalent of one standard deviation is defined as:

$$s = \frac{a}{\sqrt{6}}$$

4.4 Step 4: Convert Quantities to Standard Uncertainties

Standard uncertainty is simply the measurement uncertainty expressed as a standard deviation. All statistically calculated uncertainty components (*Type A*, *Type B*- Rectangular Distribution, and *Type B*- Triangular Distribution) should already be expressed as one standard deviation.

For an uncertainty component that is being evaluated and quantified as *Type B* from a calibration certificate (or other information source), the certificate (or information source) must be carefully reviewed in order to arrive at the standard uncertainty. For example, calibration certificates generated by NIST are typically calculated assuming a normal distribution and reported at a 95% confidence level (k = 2). In this case, the reported uncertainty on the certificate will be divided by the coverage factor, 2, to arrive at the standard uncertainty.

In preparation for the next step, all standard uncertainties must be expressed in the same measurement unit. If the same measurement unit is not associated with each standard uncertainty, then convert each standard uncertainty into a percentage (i.e., relative standard

uncertainty).

4.5 Step 5: Calculate Combined Standard Uncertainty

In this step, all of the individual standard uncertainties are combined to calculate a standard uncertainty of the measurement process, which is an estimated standard deviation. This combined standard uncertainty [$u_c\ (y)$] is calculated as the positive square root of the variance of all the combined uncertainty components:

$$u_c(y) = \sqrt{s_p^2 + u_0^2 + u_1^2 + u_2^2 + \ldots\ldots + u_i^2}\ ,$$

where s_p is the *Type A* calculated standard uncertainty for the measurement process and u_i are the *Type B* calculated standard uncertainties.

4.6 Step 6: Expand the Combined Standard Uncertainty by Coverage Factor (*k*)

The combined standard uncertainty calculated in the previous step is an estimated standard deviation with a confidence level of 68.27% ($k = 1$). In the CU, the combined standard uncertainty will be expanded by an appropriate coverage factor (k) to yield a confidence level of ≥99.7%. The specific value for the coverage factor is based on the amount of data that is available for the measurement process (i.e., *Type A* data). Table 1 provides the coverage factor (k) to apply based on the degrees of freedom (n-1), where n is equal to the number of *Type A* data points. Coverage factors other than those shown in Table 1 can be calculated using the TINV function in Excel®. The combined standard uncertainty [$u_c\ (y)$] is simply multiplied by the coverage factor to yield the expanded uncertainty (U) as shown below:

$$U = k * u_c(y)$$

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	A	B	C
1	**Degrees of Freedom (*n*-1)**	**Probability**	***k*-value (99.73% CL)**
2	1	0.0027	235.7837
3	2		19.2060
4	3		9.2187
5	4		6.6201
6	5		5.5070
7	6		4.9040
8	7		4.5299
9	8		4.2766
10	9		4.0942
11	10		3.9569
12	11		3.8499
13	12		3.7642
14	13		3.6941
15	14		3.6358
16	15		3.5864
17	16		3.5441
18	17		3.5075
19	18		3.4754
20	19		3.4472
21	20		3.4221
22	25		3.3296
23	30		3.2703
24	35		3.2291
25	40		3.1987
26	45		3.1755
27	50		3.1571
28	100		3.0767
29	∞		3

4.7 Step 7: Evaluate the Expanded Uncertainty

In this step the expanded uncertainty (U) is evaluated to determine if it "makes sense" and is "reasonable". This evaluation may identify calculation errors that can be corrected. Additionally, if pre-determined "acceptable limits" were defined for measurement uncertainty, then the expanded uncertainty should be evaluated against the "acceptable limits". If the measurement uncertainty is deemed to be unacceptable, areas of method improvement can be identified and evaluated for their impact on the estimation of measurement uncertainty using the information available from Steps 3 and 4.

4.8 Step 8: Report the Uncertainty

Expanded uncertainty will be rounded up and reported with two or less significant figures. This rounding up should only be done at the end of the measurement uncertainty calculation, to prevent cumulative effects from rounding up each standard uncertainty value. The reported measurement result will be truncated to the same level of significance that the rounded expanded uncertainty is reported. For example, if the measurement uncertainty of methamphetamine concentration in blood is 29 ng/mL (99.7% confidence level), and the measurement result for the case specimen is 498.23 ng/mL, then the measurement result will be truncated and reported as 498 ng/mL.

When reporting quantitative values in a *Laboratory Report*, the CU will include the measurement result with the associated expanded uncertainty and the confidence level. For example, a report may state that "Ethanol was identified in the Item 1 blood specimen at a concentration of 0.19 ± 0.03 gram % (99.7% confidence level)."

5 References

Joint Committee for Guides in Metrology (JCGM), *Evaluation of measurement data- Guide to the expression of uncertainty in measurement* (GUM) (GUM 1995 with minor corrections). (Sevres, France: International Bureau of Weights and Measures [BIPM]-JCGM 100, September 2008). Available at http://www.bipm.org/en/publications/guides/gum.html.

National Institute of Standards and Technology, *SOP 29 – Standard Operating Procedure for the Assignment of Uncertainty*, (Gaithersburg, Maryland, February 2012). Available at http://www.nist.gov/pml/wmd/labmetrology/upload/SOP_29_20120229.pdf.

Rev. #	Issue Date	History
5	02/09/18	Defined "NIST" in section 1, used acronym in remainder of document. Edited section 2 to include applicable personnel and to align language with ASCLD/LAB policy and QAM. Changed "document" and related to "record" and related in section 3. Edited last paragraph in section 4 to remove specific Excel reference. Removed "personal bias" from last bullet in section 4.2. Replaced "subunit" with "CU" in section 4.3.1.1. Changed confidence level in section 4.6 from 99.73% to ≥ 99.7%. Changed to "*Laboratory Report*" in section 4.8 and changed "Q1" to "Item 1".
6	09/13/19	Removed reference to ASCLD/LAB guidance documents in section 1 and references section. Expanded the scope in section 2 to all reported quantitative results. Added "intermediate precision" and reference to "coverage probability" in section 3. Removed "subunit" in sections 3 and 4.3.1.1, replaced with "standard operating procedures" in section 4.3.1.1.

Approval

Redacted - Signatures on File

General Chemistry
Technical Leader: Date: 09/11/2019

Acting Toxicology
Technical Leader: Date: 09/11/2019

Metallurgy
Technical Leader: Date: 09/11/2019

Paints and Polymers
Technical Leader: Date: 09/11/2019

Chemistry Unit Chief: Date: 09/11/2019

QA Approval

Quality Manager: Date: 09/11/2019

Chemistry Unit
Procedures for the Procurement of Services and Supplies

1 Purpose

During its day-to-day operations, the Chemistry Unit (CU) uses a variety of services and supplies. These procedures describe the steps for purchasing services and supplies within the Unit.

2 Scope

This document applies to CU personnel involved in the procurement of services and supplies.

3 Procurement of Services and Supplies

Qualified technical staff will prepare the *Requisition for Supplies and Equipment* (FD-369), or equivalent, for all services and supplies. All requests for services and supplies will be approved by the Unit Chief prior to ordering. When ordering a reference material(s), a copy of the FD-369 (or equivalent) will be provided to the CU's Chemical Inventory Manager (CIM). This will alert the CIM that a reference material has been ordered.

4 Records

The receipt of all supplies or services will be recorded by the person receiving the order. When items are received, they will be checked against the original FD-369, or equivalent, to ensure that they are in agreement with one another. If they are, the packaging records supplied with the order will be marked to indicate the items were received, then initialed and dated. Purchasing records will be retained electronically by the CU purchase card holders in the Enterprise Process Automation System (EPAS).

5 Storage of Supplies

Supplies will be stored in appropriate storage locations. Specialized storage conditions, as defined by the manufacturer of the item, will be met.

6 Evaluation of Critical Suppliers

A new supplier of critical consumables, supplies, and services will be evaluated upon first use. The *Chemistry Unit Critical Supplier Assessment Form* (Appendix A) will be used to record the evaluation. The completed forms will be maintained within the CU.

A list of approved suppliers of critical consumables, supplies, and services is maintained on the CU share drive (S:\Cushare). If a critical supplier, whether new or already approved, consistently fails to meet the requirements of the CU, a new critical supplier will be identified and evaluated. A critical supplier that demonstrates a history of unacceptable performance will be removed from the approved suppliers list.

Rev. #	Issue Date:	History:
3	02/04/14	Deleted second sentence in section 3. Revised section 6- changed "distributor" to "critical supplier", removed "historical performance" evaluation criteria, removed "*New*" from title of assessment form, changed location of approved suppliers list from CU share drive to CHEMNET. Revised *CU Critical Supplier Assessment Form* (Appendix A).
4	02/09/18	Revised section 2 to include personnel. Changed "reviewed by" to "approved by" in section 3 and removed "or designee" from "Unit Chief or designee". Changed title of section 4 and added used of EPAS for record storage; replaced "document" with "record" (or similar) throughout. Updated storage location of approved suppliers in section 6.

Approval

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General Chemistry
Technical Leader: Date: 02/08/2018

Toxicology
Technical Leader: Date: 02/08/2018

Metallurgy
Technical Leader: Date: 02/08/2018

Paints and Polymers
Technical Leader: Date: 02/08/2018

Chemistry Unit Chief: Date: 02/08/2018

QA Approval

Quality Manager: Date: 02/08/2018

Appendix A: *CU Critical Supplier Assessment Form*

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Chemistry Unit
Procedures for Measurement Traceability

1 Purpose

Measurement traceability, formally referred to as metrological traceability, is defined in the VIM (JCGM, *International vocabulary of metrology*) as "a property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, each contributing to the measurement uncertainty". Measurement traceability can be characterized by the following essential elements:

- Unbroken Chain of Comparisons
- Documented Measurement Uncertainty
- Documented Measurement Procedure
- Technical Competence
- Realization of the International System of Units (SI Units)
- Documented Calibration Intervals
- Measurement Assurance

Several of these elements (e.g., documented measurement uncertainty, documented measurement procedure, technical competence) are addressed in other FBI Laboratory and/or Chemistry Unit (CU) quality system documents.

2 Scope

This document applies to CU personnel recording and/or reporting measurement results that require an estimation of measurement uncertainty. These measurements are defined in the *CU Procedures for Estimating Measurement Uncertainty*. Measurement traceability is required for all measurements where measurement uncertainty is estimated.

3 Establishing Measurement Traceability

3.1 Establishing Measurement Traceability Through the Calibration of Equipment Used

3.1.1 Equipment List

The following CU equipment requires calibration when the measurement accuracy or measurement uncertainty of the equipment affects the validity of the examination and/or the

calibration is required to establish metrological traceability of the examination. This list is not exhaustive. If other applicable equipment exists it will be addressed in the appropriate CU standard operating procedure(s).

- Balances
- Mass Reference Standards
- Pipettes
- Rockwell Hardness tester (HRB and HRC scales)
- Microhardness tester (Knoop and Vickers scales)
- Micrometers
- Calipers
- Gauge blocks
- Load cells
- Extensometers
- SmartScope

The following CU equipment does not require calibration, as the equipment calibration has been demonstrated to not be significant to the measurement result and associated measurement uncertainty:

- Volumetric glassware (e.g., volumetric flasks)

3.1.2 Calibration of Equipment

Suppliers of external calibration services are used for the calibration of CU's equipment. The calibration due date is maintained in Resource Manager in Forensic Advantage (FA) and is indicated on (or near) the equipment. The applicable CU standard operating procedures and the certificates provided by the external calibration companies contain relevant information to include:

- Calibration service provider specifications
- Calibration requirements
- Calibration interval
- Intermediate checks of calibration status ('Calibration Check')

3.2 Establishing Measurement Traceability Through Reference Materials

3.2.1 Calibrators

Certified reference materials (CRM) with valid measurement traceability will be used as the source of calibrators when calibrators are used in conjunction with a measuring system to

establish measurement traceability. If the CRM is changed in a way that alters the traceable measurement value (e.g., dilution) then calibrated equipment used to alter the CRM (e.g., pipette) will be considered part of the traceability chain.

If a valid CRM is not available, then the reference material used to prepare a calibrator must be evaluated and its competence and traceability confirmed. Objective evidence of the confirmation will be recorded.

Specific information related to preparation and evaluation of calibrators can be found in the applicable CU standard operating procedures.

4 Measurement Assurance

Applicable CU standard operating procedures contain information on the checks utilized to maintain confidence in the calibration status of the equipment and reference materials used for measurements.

5 Documentation

Documentation of measurement traceability relies upon a variety of records. Technical Leaders of categories of testing that require demonstration of measurement traceability will ensure the necessary records are compiled into a measurement traceability file, which may consist of paper and/or electronic records. At a minimum, this measurement traceability file will contain:

- A list of uncertainty components deemed to be significant to the measurement and how traceability of each of the uncertainty components is established (i.e., through calibration of equipment or through reference materials).
- Copies of relevant external calibration certificates, or an indication that the certificates exist and where they are located.
- Supporting documentation that demonstrates an external calibration company meets the requirements indicated in the *FBI Laboratory Practices for the Calibration and Maintenance of Equipment*, or an indication where the documentation is located.
- Supporting data and/or calculations that demonstrate calibration of particular equipment is not significant to the measurement result and associated measurement uncertainty (where applicable).
- Copies of relevant CRM certificates, or an indication that the certificates exist and where they are located.
- Supporting documentation that demonstrates a CRM provider meets the requirements indicated in the *FBI Laboratory Practices for the Calibration and Maintenance of*

Equipment, or an indication where the documentation is located.

- Objective evidence of the confirmation of calibrators prepared from a reference material that was not accompanied with valid measurement traceability (i.e., non-CRM). Refer to the *CU Procedures for Verification of Reference Materials* for approaches to identity and purity verification.

6 References

Joint Committee for Guides in Metrology (JCGM), *International vocabulary of metrology – Basic and general concepts and associated terms* (**VIM**), 3rd ed. (Sevres, France: International Bureau of Weights and Measures [BIPM]-JCGM 200, 2012) (2008 with minor corrections).

Chemistry Unit Quality Assurance and Operations Manual- Procedures for Estimating Measurement Uncertainty

Chemistry Unit Quality Assurance and Operations Manual- Procedures for Verification of Reference Materials

Toxicology Standard Operating Procedures Manual

Metallurgy Standard Operating Procedures Manual

Instrument Operation & Support Standard Operating Procedures Manual

Instrument Operation and Support System (electronic database maintained by IOSS)

Joint Committee for Guides in Metrology (JCGM), *Evaluation of measurement data- Guide to the expression of uncertainty in measurement* (GUM) (GUM 1995 with minor corrections). (Sevres, France: International Bureau of Weights and Measures [BIPM]-JCGM 100, September 2008).

National Institute of Standards and Technology, *SOP 29 – Standard Operating Procedure for the Assignment of Uncertainty*, (Gaithersburg, Maryland, February 2012).

Rev. #	Issue Date	History
1	02/09/18	Edited section 2 to include applicable personnel and to align language with ASCLD/LAB policy and QAM. Removed "subunit" where applicable. Edited opening paragraph and added equipment to list in section 3.1.1. Added reference to Resource Manager in FA in section 3.1.2. Deleted paragraph that was in section 3.2, moved relevant content to section 3.2.1. Removed specific references in section 3.2, change to 'applicable CU SOPs' (too many to specify and subject to change). Fixed typos in section 3.2. Deleted sections 3.3, 4.1, and 4.2 and edited section 4 to refer to the applicable CU SOPs. Edited language in section 5 for clarity. Deleted specific CU SOPs from section 6 and replaced with reference to the overall SOP manuals.
2	09/13/19	Deleted last sentence in section 1. Removed the specific measurements that require uncertainty from section 2 and referred to *CU Procedures for Estimating Measurement Uncertainty* document to eliminate redundancy. Edited section 3.1.1 to clarify that only equipment of the type listed that is used for significant measurements/measurement assurance requires calibration; also edited Rockwell tester, added microhardness tester, and added extensometers (removed strain gauges) to bulleted list. Clarified that 'calibrated' used to alter a CRM will be considered part of the traceability chain in section 3.2.1. Changed section 5 to refer to *FBI Laboratory Practices for the Calibration and Maintenance of Equipment*. Removed website addresses from references since one or more addresses had changed with time. Removed ASCLD/LAB guidance documents from references.

Approval

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General Chemistry
Technical Leader: Date: 09/11/2019

Acting Toxicology
Technical Leader: Date: 09/11/2019

Metallurgy
Technical Leader: Date: 09/11/2019

Paints and Polymers
Technical Leader: Date: 09/11/2019

Chemistry Unit Chief: Date: 09/11/2019

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Quality Manager: Date: 09/11/2019