Developing and Validating Technical and Chemical Procedures

1 Purpose

This document sets forth the procedures for method development and validating technical and chemical procedures and supplements the requirements of the FBI Laboratory *Quality Assurance Manual (QAM)* and the FBI Laboratory *Operations Manual (LOM)*.

2 Scope

These procedures apply to caseworking personnel conducting explosives chemistry and explosives and hazardous devices analyses who develop new methods and validate new technical and chemical procedures.

3 Procedures

3.1 Method Development

Validation starts after a method is acquired or developed. If a method needs to be developed in the Explosives Unit (EU),including the modification of an acquired method, the method development will be a planned activity. Method development is the acquisition and evaluation of test data for the determination of conditions and/or limitations of a novel method to achieve consistent results.

Method development plans will be recorded and approved according to the *LOM Practices for Developing Methods and Validating Technical Procedures*. The *Explosives Development and Validation Plan and Review Form* (Appendix A) will be used to record the method development plan and review.

3.2 Validation Study

Validation is the process for determining whether specified requirements are adequate for intended use. The validation of an analytical procedure is referred to as a validation study in the EU. 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.

A validation plan for a technical or chemical procedure will be recorded and evaluated for approval according to the *LOM Practices for Developing Methods and Validating Technical Procedures*. A validation study for a chemical procedure must additionally meet the requirements outlined in the *LOM 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 (e.g., physical property measurements) will be limited to the accuracy as listed in section 3.2.1 of this document.

3.2.1 Performance Characteristic for Measurement of a Physical Property

Materials may be identified by their physical properties and composition. A physical property of a material can be measured without changing the composition or identity of the material (e.g., density).

3.2.1.1 Reliability

Accuracy is the closeness of an analytical result to its true, correct, or accepted value. Precision is the closeness of repeated measurements to each other. The implementation of a method that reduces systematic and random errors will improve reliability (accuracy and precision). When appropriate, measurement results should be based on the average of more than one measurement.

The accuracy of a physical property measurement can be determined by comparison of the measurement result with the true value. At a minimum, ten (10) 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.

3.3 Method Development and Validation Review and Records

Method development plans and/or validation studies including a validation summary will be recorded and reviewed according to the *LOM Practices for Developing Methods and Validating Technical Procedures*. The approvals by the Unit Chief(s) and appropriate Technical Leader will be recorded on the *Explosives Development and Validation Plan and Review Form* and when applicable, *Validation of Chemical Procedures Review Form* (7-267). The completed review form(s) will be maintained with the validation file.

Previously validated procedures that will be used in a new facility will be approved according to the *LOM Practices for Developing Methods and Validating Technical Procedures*.

In extreme situations (e.g., court mandates) when a validated procedure must be used prior to being formally written and reviewed (i.e., issued), 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 clear, written documentation of the steps that were taken to generate the results. In these instances, at a minimum, the validation data will be technically reviewed by the appropriate Technical Leader prior to using

the validated procedure for casework. This will be treated as a minor or major deviation, as appropriate, according to the *LOM Practices for Authorizing Deviations*.

Method development and validation records will be maintained within the validation file (e.g., on unit bookshelves, in electronic format, in the related case notes with respect to case-specific validation).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 a standard operating procedure (SOP). In these instances, the following criteria will be met:

- A validation plan will be created and approved by the appropriate Technical Leader using the *Explosives Development and Validation Plan and Review 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 reviewed and approved by the appropriate Technical Leader and Unit Chief(s). This will be recorded on the *Explosives Development and Validation Plan and Review Form* and when applicable, the *Validation of Chemical Procedures Review Form*.
- A copy of the review form(s) 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 in a central location in the validation file.
- If the procedure is performed routinely, an SOP will be prepared or it will be incorporated into an exisiting SOP. Required reviews and approvals will be obtained before issuance of the new procedure.

3.4 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. Competency tests are not required when new instrumentation is implemented for the same purpose. This test will demonstrate that applicable personnel can accurately perform the procedure stated in the SOP. For personnel that were involved in the validation process, the Unit Chief(s) and the 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 in lieu of a competency test, will be recorded in the employee's training records.

3.5 Procedure Modifications

Modifications and/or deviations from a procedure will be authorized and recorded according to the *LOM Practices for Authorizing Deviations*. The *LOM Practices for Validating Chemical Procedures* will be followed when procedures are modified from validated chemical procedures.

3.6 Minor Deviations

To maintain consistency when other personnel are faced with the same or similar analyses, all minor deviations to SOPs will be recorded in accordance with the *LOM Practices for Authorizing Deviations*. At a minimum, the record will include the 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, and the name and initials or the signature of the approver and the date of the authorization. Additionally, the FBI Laboratory number associated with the minor deviation, the analyte(s) that were targeted, the date of the minor deviation, the personnel that performed the minor deviation, the personnel that approved the minor deviation, the name of the procedure affected, and any additional relevant information will be included. This will allow for centralized review of minor deviations on an annual basis without accessing case files.

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.

Rev. #	Issue Date	History
3	12/16/2019	Removed SAU Chief from approval lines. Updated validation definition to comply with LOM. Added to section 3.1 and 3.2 to distinguish method development and validation.
4	07/15/2020	Removed fire debris from section 2 and removed Fire Debris Technical Leader from approval lines. Added definitions to section 3.2.

Redacted - Signatures on File

Approval

Explosives Chemistry Technical Leader — Date: 07/14/2020

Explosives and Hazardous Devices Technical Leader — Date: 07/14/2020

Explosives Unit Chief — Date: 07/14/2020

QA Approval

Quality Manager — Date: 07/14/2020

Appendix A: *Explosives Development and Validation Plan and Review Form*

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