

Validation in ISO 17025 Accredited Laboratories – Policy Guidance and a Recent Example of a Validation Study

INTERNATIONAL ASSOCIATION FOR IDENTIFICATION
Atlanta, GA

Robert Ramotowski



*U.S. Department of
Homeland Security*

United States
Secret Service

Disclaimer

The views expressed in this article are those of the authors and do not necessarily represent the views of the United States Secret Service or the United States government. References to a specific manufacturer or product are for information purposes only and do not imply endorsement by the authors, their employers, or the United States government.



*U.S. Department of
Homeland Security*

United States
Secret Service

ISO/IEC 17025:2005(E) Requirements



*U.S. Department of
Homeland Security*

United States
Secret Service

ISO/IEC 17025:2005(E)

■ Section 5.4.5.2

- The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods *to confirm that the methods are fit for the intended use.*
- *The validation shall be as extensive as is necessary to meet the needs of the given application or field of application.*
- The laboratory shall record the results obtained, the procedure used for the validation, and a statement as to whether the method is fit for the intended use.



U.S. Department of
Homeland Security

United States
Secret Service

ISO/IEC 17025:2005(E)

▪ Section 5.4.5.2 (NOTE 2)

The techniques used for the determination of the performance of a method should be one of, or a combination of, the following:

- Calibration using reference standards or reference materials
- Comparison of results achieved with other methods
- Inter-laboratory comparisons;
- Systematic assessment of the factors influencing the result;
- Assessment of the uncertainty of the results based on scientific understanding of the theoretical principles of the method and practical experience.



*U.S. Department of
Homeland Security*

United States
Secret Service

ISO/IEC 17025:2005(E)

■ Section 5.4.5.4

- Prior to implementation of a validated method new to the laboratory, the reliability of the method shall be demonstrated in-house against documented performance characteristics of that method.
- Records of performance shall be maintained for future reference.



*U.S. Department of
Homeland Security*

United States
Secret Service

ISO/IEC 17025:2005(E)

▪ Section 5.4.7.2

When computers or automated equipment are used for the acquisition, processing, recording, reporting, storage or retrieval of test or calibration data, the laboratory shall ensure that:

- a) *Computer software developed by the user is documented in sufficient detail and is suitably validated as being adequate for use;*

NOTE: Commercial off-the-shelf software (e.g., word processing, database and statistical programs) in general use within their designed application range may be considered to be sufficiently validated. *However, laboratory software configuration/modifications should be validated as in 5.4.7.2a.*



U.S. Department of
Homeland Security

United States
Secret Service

Nomenclature

- Level I Validation
 - Used for novel techniques (or major modifications of an existing technique) or pieces of equipment. Requires extensive testing of most of the key elements and documentation.
- Level II Validation
 - Used for minor modifications to existing techniques; software modifications; evaluation of COTS equipment. Requires approximately 50-100 samples and documentation.
- Level III Validation (modified function/performance test)
 - Used for equipment that takes no measurements or collects any analytical data (e.g., cameras, imaging systems, light sources). Requires only 10-25 samples and documentation.



*U.S. Department of
Homeland Security*

United States
Secret Service

Nomenclature

- Exemptions
 - It should be noted that certain equipment, like purely optical devices (e.g., magnifiers, stereomicroscopes), do not require validation testing or documentation. This would also apply to other equipment like scanners, optical microscopes, and commercial-off-the-shelf software packages (e.g., Microsoft Office, Photoshop). This type of equipment need only be tested to ensure that it is in good working order and no formal documentation of performance is required.



*U.S. Department of
Homeland Security*

United States
Secret Service

Key Elements Validation Level I



*U.S. Department of
Homeland Security*

United States
Secret Service

Key Elements – Level I

- The following elements should be considered (but not all of them need be addressed):
 - Accuracy – agreement between accepted and obtained values.
 - Precision – consistency of measurements.
 - Range – upper/lower limits of detection (e.g., split depleted LP).
 - Repeatability – intra-assay precision.
 - Reproducibility – replication of data by another examiner.
 - Robustness – efficacy of method to small variations in parameters.
 - Specificity – ability to detect analyte in presence of other components.

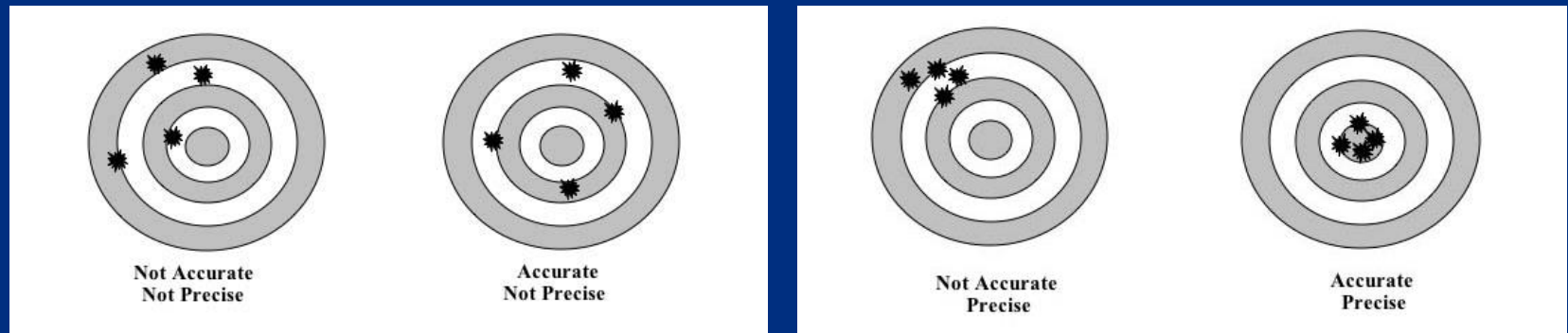


*U.S. Department of
Homeland Security*

United States
Secret Service

Accuracy/Precision

- Accuracy is the agreement between the accepted and the obtained value.
- Precision is the ability of a measurement to be consistently reproduced.



http://celebrating200years.noaa.gov/magazine/tct/accuracy_vs_precision.html (accessed 6/27/14)

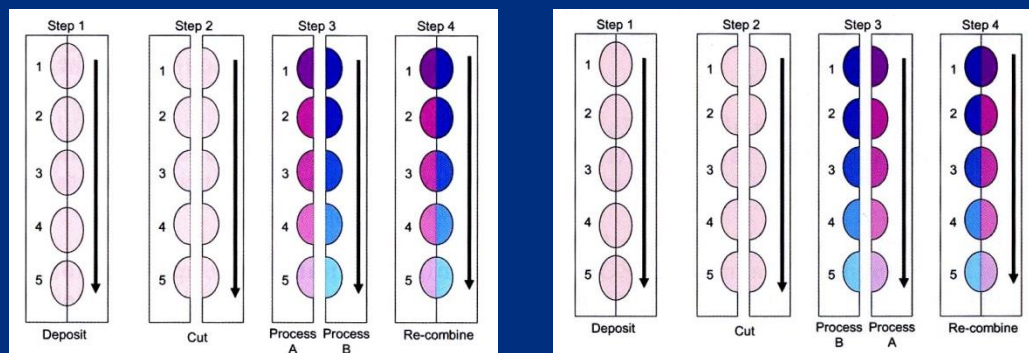


*U.S. Department of
Homeland Security*

United States
Secret Service

Range

- Range covers the upper and lower values of a particular analyte in a sample capable of being detected by a method.
 - (e.g., the use of split depletion samples to create a range of amino acids or lipid concentrations to test amino acid or lipid reagents).



U.S. Department of
Homeland Security

United States
Secret Service

From: Lee JL, Bleay SM, Sears VG, Mehmet S, Croxton R. Evaluation of the Dimethylamino-cinnamaldehyde Contact Transfer Process and its Application to Fingerprint Development on thermal Papers. *J Forensic Ident.* 2009;59(5):551.

Repeatability

- Repeatability – intra-assay precision; measurements by one person or instrument on the same item (and over a short time interval).
 - Can one examiner using a particular instrument (e.g., GC-MS) or method (e.g., ninhydrin) process the same sets of samples on different days and obtain the same (or similar) results that are acceptable?



*U.S. Department of
Homeland Security*

United States
Secret Service

Reproducibility

- Reproducibility is the ability of a result to be replicated by someone else independently.
 - Can multiple examiners using a particular instrument (e.g., GC-MS) or method (e.g., ninhydrin) process the same sets of samples and obtain the same (or similar) results that are acceptable?
 - Can the technique be reproduced by a competent practitioner in another laboratory with the same equipment and resources?



*U.S. Department of
Homeland Security*

United States
Secret Service

Robustness

- Robustness – The resistance to small variations in method parameters.
 - Use of multiple substrate types.
 - Use of multiple donors/samples.
 - Changes in environmental conditions (e.g., temperature, %RH).
 - Changes in concentrations of certain components of a method (e.g., changing the concentration of ferric nitrate in PD to see if it changes the expected result).



*U.S. Department of
Homeland Security*

United States
Secret Service

Specificity

- Specificity – The ability to assess an analyte in the presence of other components.
 - Does the method successfully develop the latent print without developing the background substrate as well (e.g., using powder suspensions on methacrylate-based adhesives)?
 - Does the presence of interfering species cause the reagent to become less effective or even ineffective (e.g., the presence of calcium ions on paper causes the reagent physical developer to bind indiscriminately)?



*U.S. Department of
Homeland Security*

United States
Secret Service

Key Elements Validation Levels II/III



*U.S. Department of
Homeland Security*

United States
Secret Service

Key Elements – Levels II/III

- **Remember – Validation Levels II/III ≠ Empirical Research**
 - Previously tested methods or pieces of equipment (i.e., COTS) that have been validated/tested or published in peer reviewed publications do not require extensive testing/experimentation.
 - These validation tests can focus primarily on repeatability testing; however, in rare cases (e.g., satellite laboratories), reproducibility would also have to be addressed.
 - Where applicable, the use of stock “test sets” to test software (e.g., ULW, FISH) can significantly increase efficiency when conducting these types of validation tests.



*U.S. Department of
Homeland Security*

United States
Secret Service

Documentation

- The Laboratory Research Proposal Form formally initiates the research process.
- Accompanied by: 1) design of experiment(s), 2) detailed cost estimate, and 3) literature review.
- The Method/Equipment Validation Form completes the formal process with approvals and impact on laboratory SOPs.



U.S. Department of
Homeland Security

United States
Secret Service

FSD Laboratory Research Proposal Form	
INSTRUCTIONS	
1. A thorough literature search of all Forensic Services Division library resources is required before submitting the request. 2. Thoroughly complete all fields contained in this document. 3. Submit completed forms to the appropriate Branch Chief to receive initial approval. 4. The Research Section staff will evaluate all research requests for scientific and technical feasibility and make the appropriate recommendation to the Laboratory Director. 5. Upon the approval of the Laboratory Director, the project may begin and resources will be applied accordingly.	
PROJECT INFORMATION	
Project Title:	
Requestor(s):	Request Date:
Tracking No.:	
Objective(s):	
Experimental Approach:	
Describe any past research in this area and include literature search results:	
Health and Safety Impact:	
PROJECT RESOURCES	
Laboratory supplies/equipment needed (beyond current laboratory resources):	
Estimated cost of additional resources:	
Will intern or contractor support be needed for this project? <input type="checkbox"/> Yes <input type="checkbox"/> No	
Estimated period of performance for this project:	
APPROVAL	
Any response other than "Approved" will require an explanation in the Comments section.	
Branch Chief Signature:	Date
Research Section Signature:	Date
Laboratory Director Signature:	Date
Comments:	
<div><input type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected <input type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected <input type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected</div>	
FSD-037-F: FSD Laboratory Research Proposal Form Approved By: FSD Laboratory Director All Printed Copies are Uncontrolled	
First Approved: Apr 2009 Revision #: 3 Revision Effected: 9/6/2013	
Page 1 of 1	

Sample Plan with Cost Estimate

1) Analytical Procedure

Thermal Ribbon Analysis Platform (TRAP) Operating Manual Draft.

2) Specificity

N/A; analyte identity is not evaluated.

3) Accuracy

Scan at least three types of thermal ribbons using the TRAP and a desktop scanner. Crosscheck the results for accuracy. (Ribbons # 1, 2, 3)

4) Precision

a. Repeatability

- i. Have a single examiner scan the same thermal ribbon once a week for four weeks. (Ribbon # 4)

b. Intermediate Precision

- i. Have no less than four examiners independently scan the same thermal ribbon. (Ribbon #5)

c. Reproducibility

N/A; no other laboratories have such a system.

5) Detection Limits

N/A; no quantitation conducted.

6) Quantitation Limits

N/A; no quantitation conducted.

7) Linearity

N/A; no quantitation conducted.

8) Range

Ribbons of various widths and lengths will be tested under Robustness section.

9) Robustness

Use the system to scan a variety of ribbons to demonstrate robustness.

Include at least:

- A foil ribbon (Ribbon # 6)
- A label maker thermal ribbon (Ribbon # 7)
- A CR80 thermal ribbon (Ribbon # 11)
- A letter size thermal ribbon (Ribbon # 8)
- A CMYK thermal ribbon (Ribbon # 9)
- A CMYKO thermal ribbon (Ribbon # 10)

10) Published Literature and Standards

Relevant literature will be pulled mostly from the FSD library and askSam database of scientific articles.

Table 1: Paper types used in this study

Paper Type	Characteristics	Manufacturer Information
White photocopy paper	20#, recycled	
White Lined paper		
Yellow legal pad paper		

Table 2: Latent print processes used in this study

Chemical Process(es)
Indanedione-zinc (acetone/PE formulations)
Ninhydrin (acetone/PE formulations)
Physical developer
Indanedione-zinc (acetone formula) + physical developer
Ninhydrin (acetone formula) + physical developer

Table 3: Supplies needed for this study

Supplies (for ~400 samples)	Number Needed	Cost
Replacement solvents ¹ (acetone, PE, ethanol, acetic acid)		\$192.47
Replacement reagents (zinc chloride)	< 1 g	nominal
PD reagent ²	3 L	\$165.00
Magnetic Powder ³	1	\$41.00
Hair Spray (AquaNet)	12	\$42.00
Total Cost		\$440.47



U.S. Department of
Homeland Security

United States
Secret Service

Method/Equipment Validation Form

- Ensure that the results are summarized and that each participant and reviewer of the work product signs and dates the validation form.
- Reviewers should consist of (but not potentially be limited to):
 - Section/Unit supervisor
 - Subject matter experts (SMEs)
 - Laboratory Director
 - Chief/Senior Scientist (or similar position)
 - Alternates/Designees (as needed)



*U.S. Department of
Homeland Security*

United States
Secret Service

Documentation

Tracking No.:

FSD Method / Equipment Validation Form

INSTRUCTIONS

1. Complete each of the fields in this document as completely as possible. For additional guidance, please refer to the Laboratory Operations Manual, Section 21, Practices for Validation of New Procedures and Equipment.
2. The tracking number will be the same one issued by the Chief Forensic Chemist on the Laboratory Research Proposal Form (FSD-037-F).
3. Submit completed forms through all of the personnel listed in order in the "APPROVALS" section. In some circumstances, the Branch Chief and Subject Matter Expert can be the same person.

PROJECT INFORMATION

Requestor(s): _____ Request Date: _____

Tracking No.: _____

CURRENT METHOD / EQUIPMENT INFORMATION

Current Method/Equipment: _____

Description of Current Method/Equipment: _____

Description of Proposed Changes to Current Method or Equipment: _____

PROPOSED METHOD / EQUIPMENT INFORMATION

Proposed Method/Equipment Title: _____

Health and Safety Impact: _____

Executive Summary of Validation Study Results: _____

FSD-056-F: FSD Method / Equipment Validation Form
Approved By: FSD Laboratory Director
All Printed Copies are Uncontrolled

First Approved: Sep 2013
Revision #: NEW
Revision Effected: --

Page 1 of 3

Tracking No.:

VALIDATION STUDY PARTICIPANTS

Name/Title: _____	Signature: _____	Date: _____
Name/Title: _____	Signature: _____	Date: _____
Name/Title: _____	Signature: _____	Date: _____
Name/Title: _____	Signature: _____	Date: _____
Name/Title: _____	Signature: _____	Date: _____

APPROVAL

Any response other than "Approved" will require an explanation in the Comments section.

Branch Chief	Signature: _____	Date: _____	<input type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Printed Name: _____			
Subject Matter Expert	Signature: _____	Date: _____	<input type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Printed Name: _____			
Quality Assurance Manager	Signature: _____	Date: _____	<input type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Printed Name: _____			
Chief Forensic Chemist	Signature: _____	Date: _____	<input type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Printed Name: _____			
Laboratory Director	Signature: _____	Date: _____	<input type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Printed Name: _____			

Comments: _____

FSD-056-F: FSD Method / Equipment Validation Form
Approved By: FSD Laboratory Director
All Printed Copies are Uncontrolled

First Approved: Sep 2013
Revision #: NEW
Revision Effected: --

Page 3 of 3



U.S. Department of
Homeland Security

United States
Secret Service

Health and Safety Impact

- Have health and safety personnel in your organization review the impact of the new equipment or method.
- Assess the impact of any new potentially hazardous chemicals.
- Assess the impact of the new chemicals on waste disposal.
- Health and safety officer should sign off on the final documentation to confirm that this review took place.



*U.S. Department of
Homeland Security*

United States
Secret Service



UNITED STATES SECRET SERVICE
FORENSIC SERVICES DIVISION
LABORATORY OPERATIONS MANUAL

FSD-005: FSD Laboratory Operations Manual
Approved By: FSD Laboratory Director
All Printed Copies are Uncontrolled

First Approved: Oct 2007
Revision #: 44
Revision Effected: 04/17/2017

Page 1 of 84



*U.S. Department of
Homeland Security*

**United States
Secret Service**

21 Practices for Validation of Procedures and Equipment

This document provides the general practices for conducting validation studies prior to the use of technical procedures or instrumentation in the FSD Laboratory. These practices shall apply to both routine and non-routine procedures. Validation is the process by which the scientific community acquires the information necessary to assess a procedure's capability for obtaining reliable and reproducible results.

21.1 Scope

These practices shall apply to all FSD laboratory personnel who are involved in validation studies of new or novel methods and/or equipment, whether the method was developed internally or externally. It is important to note that regardless of whether the new or modified technique and/or equipment was validated externally or published in a peer-reviewed scientific journal, an internal validation study is still required before it can be applied to casework.

The scope of the validation study shall depend on whether or not the method/equipment is novel or if it involves a mature technology or procedure. Established methods or technologies that have been published in peer-reviewed scientific journals would require a more limited validation study whereas a truly novel method or piece of equipment would require a more extensive validation. The impact of the changes will dictate the scope of the validation study. Validation testing has been broken down into different levels based on what testing is required. These different levels (as well as exemptions) are explained in the subsections below. Note that all testing should be properly documented (see also QAM 5.5 Equipment or 5.5.2 and LOM 7.5 Equipment Calibration and Maintenance).

21.1.1 Level I Validation

A level I validation is a comprehensive examination of the technical and scientific parameters of a truly novel technique or instrument. This level of validation would require extensive sample testing to fully examine repeatability and reproducibility (or other factors listed in section 21.3.1.2). Examples of what would qualify for a level I validation study would include the Thermal Ribbon Analysis Platform (TRAP) system (a completely new and unique instrument) and the original introduction of 1,2-indanedione-zinc (a completely new and unique method). For guidance in developing a project plan for level I validation studies, the appropriate peer reviewed guidelines should be consulted (e.g., Sears VG, Bleay SM, Bandey HL, Bowman VJ. A Methodology for Finger Mark Research. Sci. Just. 2012, 52 (3), 145-160; International Fingerprint Research Group. Guidelines for the Assessment of Fingerprint Detection Techniques. J Forensic Ident 2014;64(2):174-200).

21.1.2 Level II Validation

A level II validation is a more limited-scale examination of the technical or scientific aspects of a well-established technique or instrument. Such an evaluation should consist of approximately 50-100 samples, require as few as two participants (to investigate repeatability and reproducibility), and be able to be completed within 1-2 weeks (depending on the complexity of the sample preparation). Examples of instruments or techniques that would require level II validation would include the replacement of the RO/DI purification system; replacement of standard, commercially available instruments (e.g., FTIR, SEM, XRF); and changing the purity or manufacture of a chemical used for processing latent prints or in a particular analytical technique. With regard to instrumentation, it would be beneficial to incorporate (if possible) a written statement from the company technician installing the equipment (along with his/her signature) that certifies that the equipment has been calibrated and is working

Silver Nitrate Validation Study



*U.S. Department of
Homeland Security*

United States
Secret Service

Robert Ramotowski

11 August 2017

26

Justification

- The international standard ISO/IEC 17025:2005(E), section 5.4.5.2, specifies that “The laboratory shall validate non-standard methods, laboratory-designed/developed methods, standard methods used outside their intended scope, and amplifications and modifications of standard methods to confirm that the methods are fit for the intended use.”
- When substituting for a chemical in any reagent, including changes in reagent grade/purity and/or manufacturer, a validation study must be conducted to ensure no loss of process efficiency will occur.
- The performance of a new chemical must be compared directly against the effectiveness of the current, validated method.



*U.S. Department of
Homeland Security*

United States
Secret Service

Background

- A chemical that meets the requirements of the U.S. Pharmacopeia and is acceptable for drug, medicinal, food, and laboratory use is labeled as USP grade.
- Chemicals labeled as technical grade are typically less pure than the other two grades. Technical grade chemicals are acceptable for industrial or commercial use, but they are not pure enough for drug, medicinal, or food use.



*U.S. Department of
Homeland Security*

United States
Secret Service

Background

- The price of silver nitrate in recent years has increased significantly, making the cost of the overall PD reagent more expensive.
- The use of a lower grade of silver nitrate could be more cost-effective; however, the potentially adverse effects of using the lower grade chemical and its potential impurities are not known.



*U.S. Department of
Homeland Security*

United States
Secret Service

FSD Laboratory Research Proposal Form

INSTRUCTIONS

1. A thorough literature search of all Forensic Services Division library resources is required before submitting the request.
2. Completely fill out all fields contained in this document.
3. Submit completed forms to the appropriate Branch Chief to receive initial approval.
4. The Research Section staff will evaluate all research requests for scientific and technical feasibility and make the appropriate recommendation to the Laboratory Director.
5. Upon the approval of the Laboratory Director, the project may begin and resources will be applied accordingly.

PROJECT INFORMATION

Project Title: Physical Developer Evaluation
 Requestor(s): Ramotowski Request Date: 4/12/11
 Objective(s):
 The objective of this project is to evaluate whether or not different grades of silver nitrate can be used to prepare physical developer. The effect of silver nitrate purity will be evaluated with respect to reagent performance.

Experimental Approach:

The experimental approach will generally follow the experimental procedure outlined in: Kent T. Standardizing Protocols for Fingerprint Reagent Testing, J Forensic Ident. 2010;60(3):371-379. Many different donors, substrates, and reagents will be used to test the various hypotheses described above. In addition, split depletion prints will be used to isolate variables for comparative evaluation.

Describe any past research in this area (include literature search results as an attachment to this request):
 n/a

FSD-037-F: FSD Laboratory Research Proposal Form
 Approved By: FSD Laboratory Director
 All Printed Copies are Uncontrolled

First Approved: Apr 2009
 Revision #: 1
 Revision Effected: 1/10/2010

Page 1 of 3

CONTINUED ON BACK

Project Title: Physical Developer Evaluation

Requestor(s): Ramotowski

Request Date: 4/12/11

PROJECT RESOURCES

Laboratory supplies/equipment needed (beyond current laboratory resources):
 n/a

Estimated cost of additional resources: n/a

Will intern or contractor support be needed for this project? ☒ Yes ☐ No

Estimated period of performance for this project: 1 month

APPROVAL

Any response other than "Approved" will require an explanation in the Comments section.

Branch Chief Signature: <i>[Signature]</i>	Date: 4/12/11	<input checked="" type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Research Section Signature: <i>[Signature]</i>	Date: 4/12/11	<input checked="" type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Laboratory Director Signature: <i>[Signature]</i>	Date: 4/12/11	<input checked="" type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected

Comments:

FSD-037-F: FSD Laboratory Research Proposal Form
 Approved By: FSD Laboratory Director
 All Printed Copies are Uncontrolled

First Approved: Apr 2009
 Revision #: 1
 Revision Effected: 1/10/2010

Page 2 of 3



U.S. Department of
 Homeland Security

United States
 Secret Service

Robert Ramotowski

11 August 2017

30

Methodology

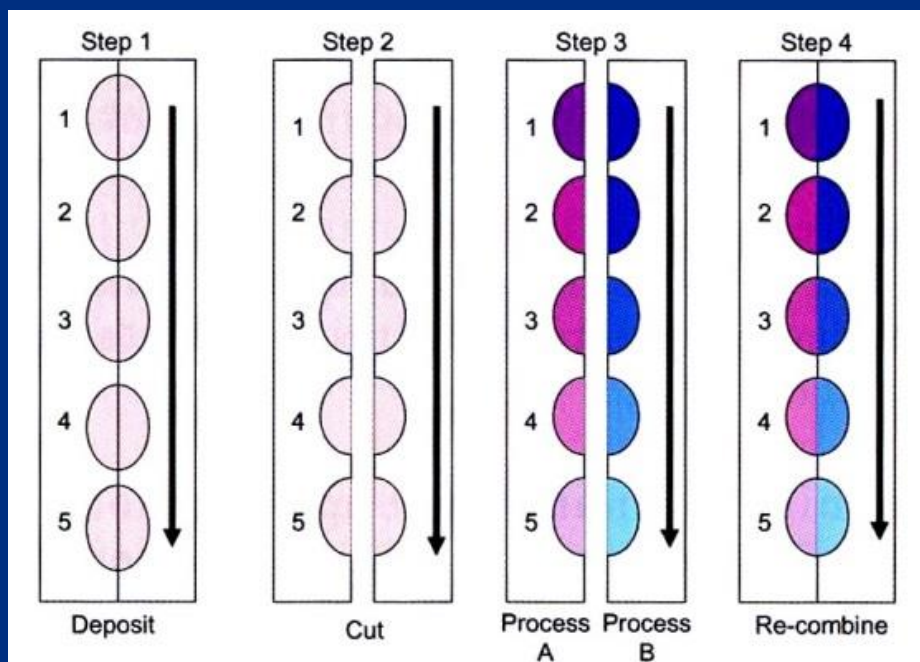
- Experiment 1: 4 females/5 males.
- 101 depletion strips (6 per strip).
- Experiment 2: 4 females/6 males.
- 101 depletion strips (6 per strip).
- Latent prints were aged from 2-12 months prior to processing.
- Evaluators were: a non-expert; 2nd year trainee; and three IAI certified examiners with 13, 23, and 30 years of experience.



*U.S. Department of
Homeland Security*

United States
Secret Service

Methodology



- Sears, V.G.; Bleay, S. M.; Bandey, H. L.; Bowman, V. J. A Methodology for Finger Mark Research. *Sci. Just.* **2012**, 52 (3), 145–160.
- International Fingerprint Working Group (IFRG). Guidelines for the Assessment of Fingerprint Detection Techniques. *J Forensic Ident* 2014;64(2):174-200.



U.S. Department of
Homeland Security

United States
Secret Service

Materials

- Substrate A: Premium white copy paper (Hammermill; 28#, 100)
- Substrate B: White photocopy paper (Xerox; 20#, 92)
- Substrate C: Steno notebook paper (Quill, 6" x 9", Gregg ruled)
- Substrate D: Newsprint (Washington Post Express)
- Substrate E: Newsprint (Washington Post)
- Substrate F: Manila envelope paper (Quill, 28#)
- Substrate G: Brown Kraft paper (Uline)



*U.S. Department of
Homeland Security*

United States
Secret Service

Evaluation/Scoring

Grade	Description
+2	Left side shows significantly better development
+1	Left side shows slightly better development
0	No difference in development
-1	Right side shows slightly better development
-2	Right side shows significantly better development

- McLaren C, Lennard C, Stoilovic M. Methylamine Pretreatment of Dry Latent Fingermarks on Polyethylene for Enhanced Detection by Cyanoacrylate Fuming. J Forensic Ident 2010;60(2):199-222.



*U.S. Department of
Homeland Security*

United States
Secret Service

PD Working Solution (Current SOP)

Redox Solution

- 30 g ferric nitrate
- 80 g ferrous ammonium sulfate
- 20 g citric acid
- 900 mL RO/DI water

Detergent Solution

- 3 g n-dodecylamine acetate
- 3 mL Tween 20
- 1 L RO/DI water

Silver Nitrate Solution

- 10 g silver nitrate
- 50 mL RO/DI water

Malic Acid Solution

- 25 g malic acid
- 1 L RO/DI water

PD Working Solution

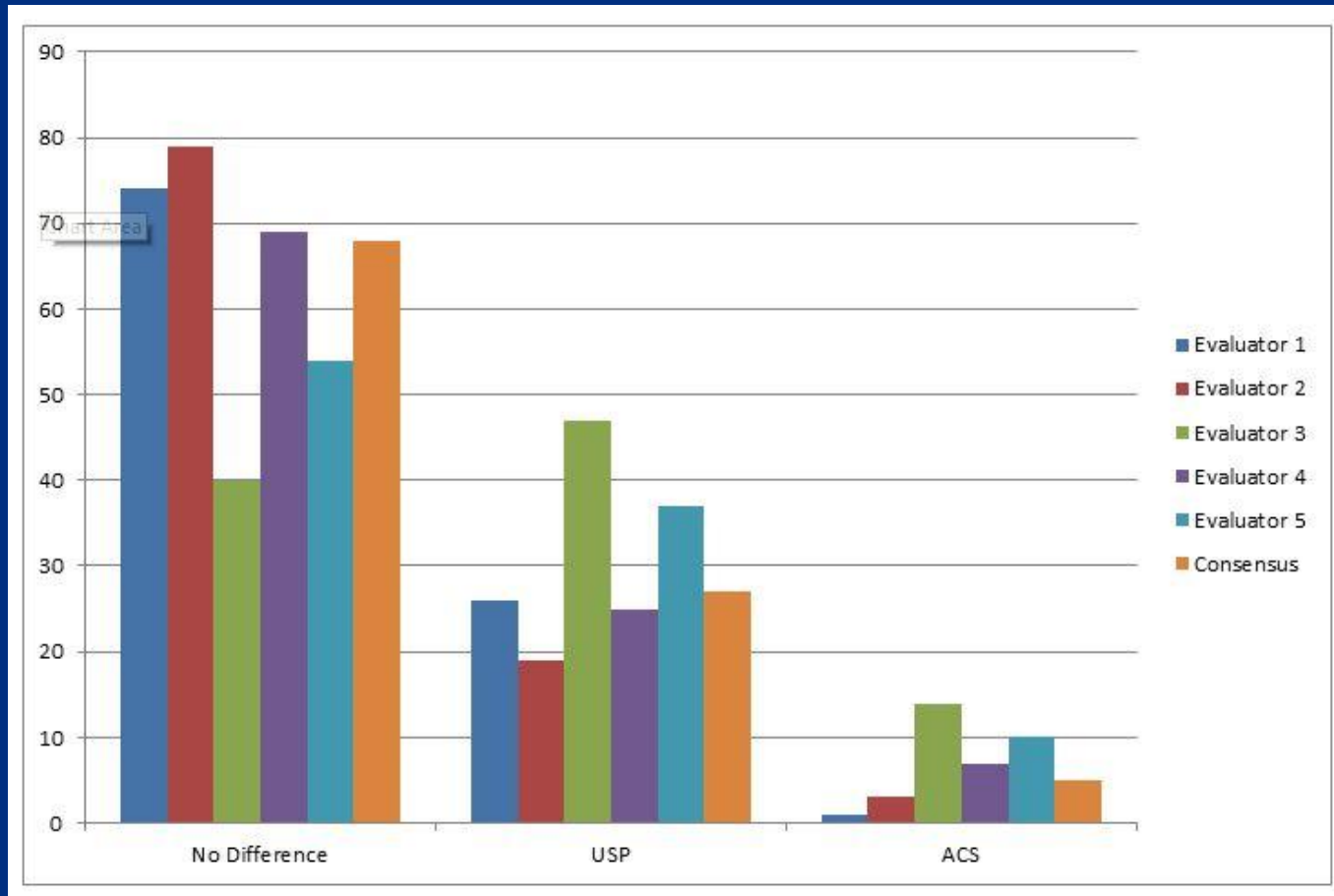
- 900 mL Redox Solution
- 40 mL Detergent Solution
- 50 mL Silver Nitrate Solution



*U.S. Department of
Homeland Security*

United States
Secret Service

Results – ACS vs. USP

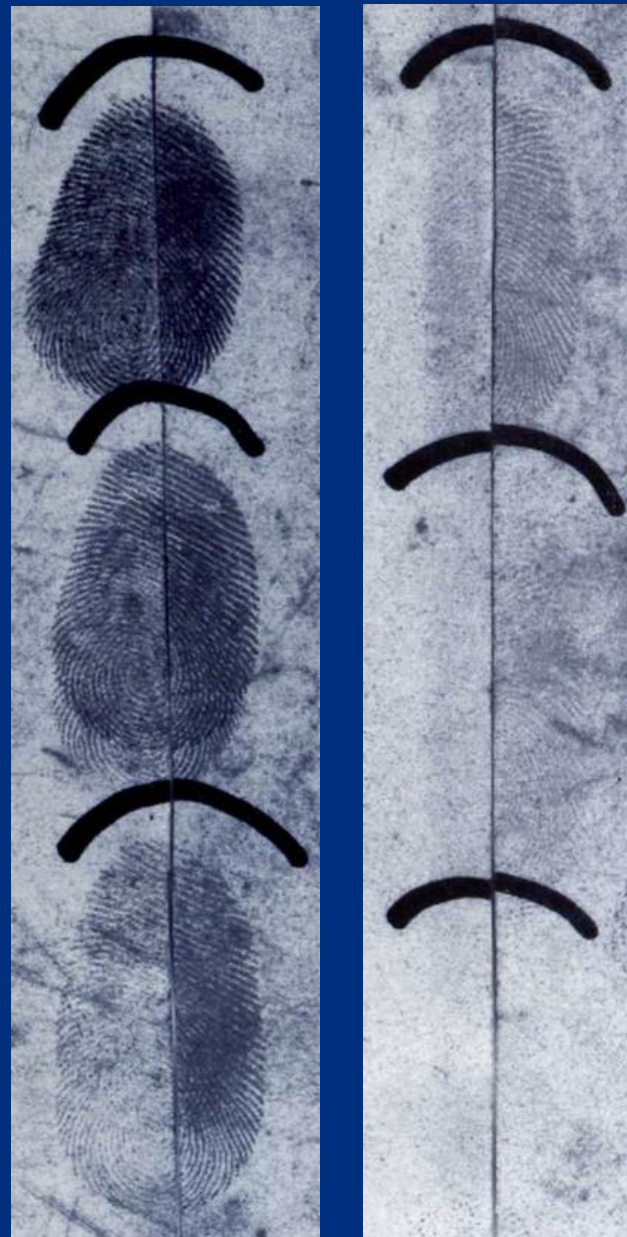


U.S. Department of
Homeland Security

United States
Secret Service

Results

- (l) An image of the first three depletions in the series of a sample from Experiment 1 in which the majority of evaluators selected the right side as being superior (the PD containing the ACS grade silver nitrate).
- (r) An image of the first three depletions in the series of a sample in which the majority of evaluators selected the right side as being superior (the PD containing the USP grade silver nitrate).



*U.S. Department of
Homeland Security*

United States
Secret Service

Results

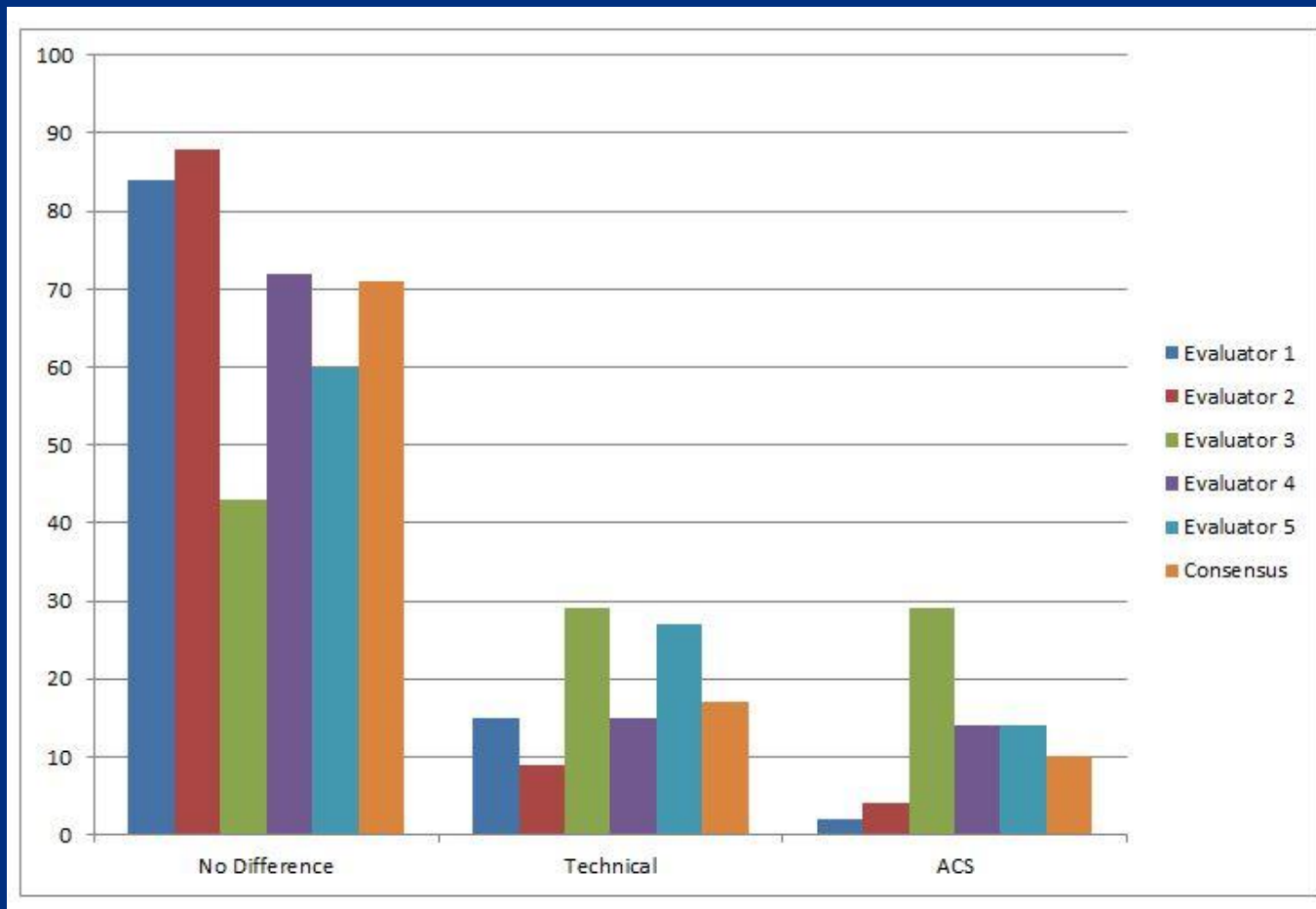
- An image of a sample from Experiment 1 that resulted in mixed voting.
- In this sample, the PD containing the ACS grade silver nitrate processed sample is on the left and the PD containing the USP grade silver nitrate processed sample is on the right.
- There was one vote for 0 and two votes each for +1 and -1.



*U.S. Department of
Homeland Security*

United States
Secret Service

Results – ACS vs. Technical



*U.S. Department of
Homeland Security*

**United States
Secret Service**

Results

- (l) An image of the first three depletions in the series of a sample from Experiment 2 in which the majority selected the right side as being superior (the PD containing the ACS grade silver nitrate).
- (r) An image of the first three depletions in the series of a sample in which the majority selected the right side as being superior (the PD containing the technical grade silver nitrate).



*U.S. Department of
Homeland Security*

United States
Secret Service

Results

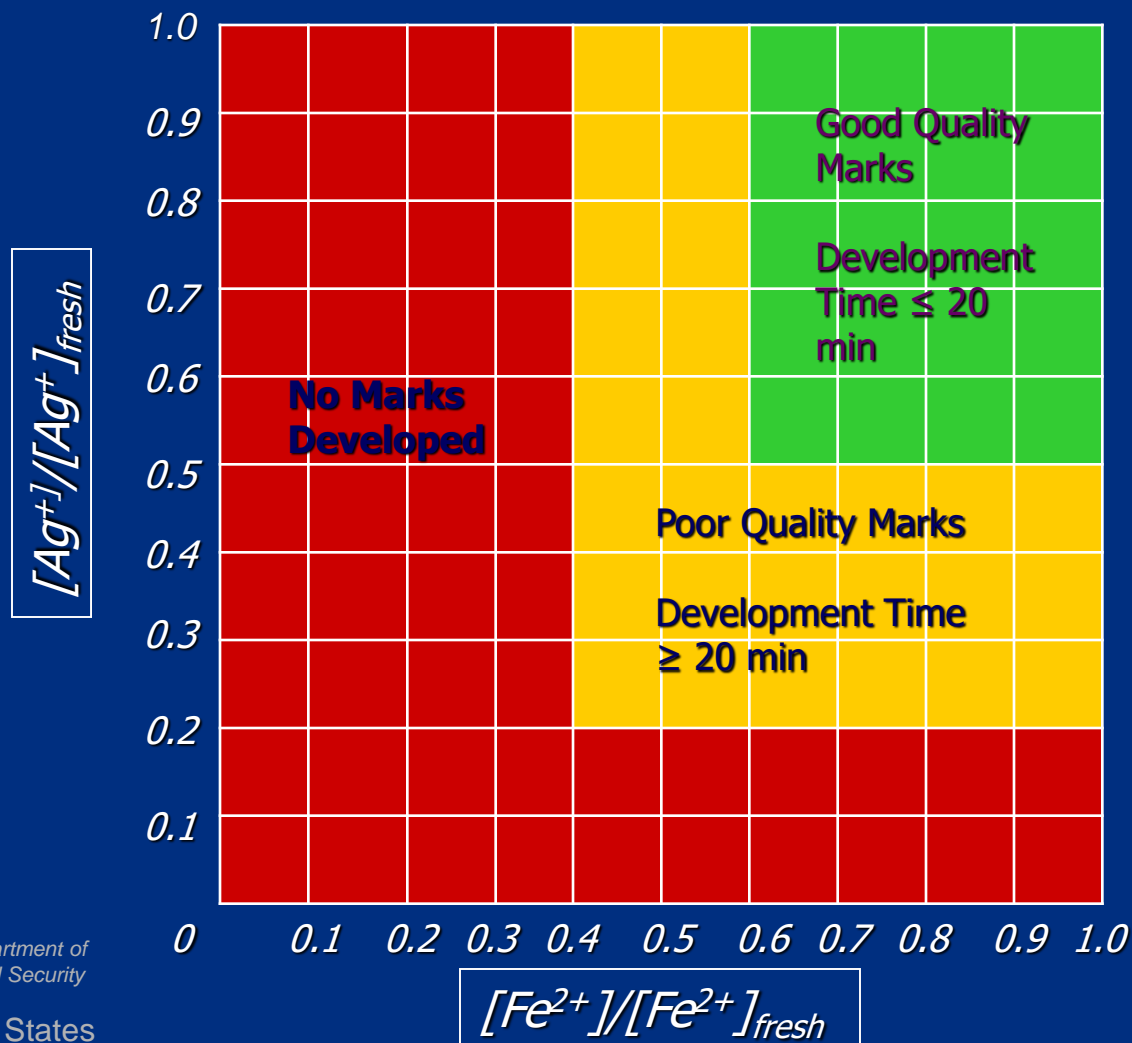
- An image of a sample from Experiment 2 that resulted in mixed voting.
- In this sample, the PD containing the ACS grade silver nitrate processed sample is on the left and the PD containing the technical grade silver nitrate processed sample is on the right.
- There was one vote for 0, two for +1, and one each for -1 and -2.



*U.S. Department of
Homeland Security*

United States
Secret Service

Discussion



U.S. Department of
Homeland Security

United States
Secret Service

FSD Method / Equipment Validation Form

INSTRUCTIONS

1. Complete each of the fields in this document as completely as possible. For additional guidance, please refer to the Laboratory Operations Manual, Section 21, Practices for Validation of New Procedures and Equipment.
2. The tracking number will be the same one issued by the Chief Forensic Chemist on the Laboratory Research Proposal Form (FSD-037-F).
3. Submit completed forms through all of the personnel listed in order in the "APPROVALS" section. In some circumstances, the Branch Chief and Subject Matter Expert can be the same person.

PROJECT INFORMATION

Requestor(s): Robert Ramotowski

Request Date: 1/25/17

Tracking No.: 2017-1

CURRENT METHOD / EQUIPMENT INFORMATION

Current Method/Equipment:
Physical Developer

Description of Current Method/Equipment:

Physical Developer is an aqueous physico-chemical method for developing latent prints on porous surfaces. The process is typically used after amino acid-specific reagents like 1,2-indanedione-zinc and ninhydrin. Its mechanism involves an oxidation reduction reaction in which an $\text{Fe}^{2+}/\text{Fe}^{3+}$ redox couple is used to reduce silver nitrate to its ground state, silver metal, via a colloidal deposition process. This metallic silver deposits on the friction ridges as dark gray or black in color due to the irregular shape of the silver particles.

Description of Proposed Changes to Current Method or Equipment:

In recent years, the cost of silver nitrate has increased dramatically and thus made the overall reagent much more expensive to use routinely in casework. As a result, a question was raised whether or not less pure grades of silver nitrate could be used to save a substantial amount of money. The proposal was to swap out the current American Chemical Society (ACS) grade of silver nitrate with two possible alternatives, United States Pharmacopeia (USP) and technical grades. These two alternative grades would be swapped out of the current formulation in a 1:1 ratio.

PROPOSED METHOD / EQUIPMENT INFORMATION

Proposed Method/Equipment Title:
Physical Developer (no change)

Health and Safety Impact:

Since the two alternative grades of silver nitrate are essentially composed of nearly entirely pure silver nitrate, there would be no additional safety and health impact over and above current practices. There would also be no impact on the current waste disposal procedures or any additional costs associated with that disposal.

Executive Summary of Validation Study Results:

This validation study examined the impact of substituting two alternative grades of silver nitrate for the current ACS grade. To investigate the impact of using a different grade of silver nitrate, a series of sebaceous-rich latent print depletion series were prepared on seven different types of paper substrates (two photocopy papers of different basis weights, two different newspaper, lined paper, manila envelope paper, and brown Kraft paper). One hundred and one depletion series strips were prepared for each comparison (ACS vs. USP and ACS vs. technical grades). All of these samples were processed with the different physical developer working solutions according to the laboratory's SOP for this reagent. The final evaluations of the developed prints were conducted by 5 volunteers, a non-expert, a 2nd year trainee, and three LAI certified fingerprint specialists with varying years of experience (13, 23, and 30 years of comparison experience).

When comparing the samples processed with PD solutions containing ACS and USP grade silver nitrate, the consensus results indicated that the ACS grade was superior approximately 5% of the time, with 27% choosing the USP grade, and 68% finding no difference.

When comparing the samples processed with PD solutions containing ACS and technical grade silver nitrate, the consensus results indicated that the ACS grade was superior approximately 10.3% of the time, with 17.3% choosing the technical grade, and 71.4% finding no difference.

It should be noted that even though a relatively small percentage of the samples were graded as being superior to the ones processed with the PD containing the current ACS grade silver nitrate, most if not all of these could still be of value.

FSD-056-F: FSD Method / Equipment Validation Form

Approved By: FSD Laboratory Director
All Printed Copies are Uncontrolled

First Approved: Sep 2013

Revision #: NEW

Revision Effected: --

Page 1 of 3

for identification purposes. Thus, even though the current ACS grade silver nitrate was chosen to produce "superior" prints 7% (versus USP) and 12% (versus technical) of the time, the overall impact of switching to either grade of these silver nitrates would be negligible. One primary reason for this is that there is a considerable excess of silver nitrate present in the PD working solution. Small changes in silver nitrate purity would have no significant impact in changing the quantity of silver ions available in solution to form the colloidal silver particles that would deposit on the latent print ridges.

The overall recommendation is that both the technical and USP grades of silver nitrate would be acceptable substitutes for the current ACS grade.

FSD-056-F: FSD Method / Equipment Validation Form

Approved By: FSD Laboratory Director
All Printed Copies are Uncontrolled

First Approved: Sep 2013

Revision #: NEW

Revision Effected: --

Page 2 of 3

U.S. Department of
Homeland SecurityUnited States
Secret Service

Robert Ramotowski

11 August 2017

43



U.S. Department of
Homeland Security

United States
Secret Service

Robert Ramotowski

11 August 2017

44

Tracking No.:

VALIDATION STUDY PARTICIPANTS		
Name/Title: Allison Fuchs/Student Intern	Signature: <i>See email for manuscript review</i>	Date: 1/26/17
Name/Title: Robert Ramotowski/Chief Forensic Chemist	Signature: <i>[Signature]</i>	Date: 1/25/17
Name/Title: Brian Jones/Fingerprint Specialist	Signature: <i>Brian A. Jones</i>	Date: 1/25/17
Name/Title: Michael Manna/Fingerprint Specialist	Signature: <i>[Signature]</i>	Date: 1/25/17
Name/Title: Esther Chervinsky/Fingerprint Specialist	Signature: <i>[Signature]</i>	Date: 1/25/17
Name/Title: Kim Smith/Fingerprint Specialist	Signature: <i>[Signature]</i>	Date: 1/25/17

APPROVAL		
Any response other than "Approved" will require an explanation in the Comments section.		
Branch Chief	Signature: <i>Brian A. Jones</i>	Date: 4/7/17 <input checked="" type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Printed Name: <i>Brian Jones</i>		
Subject Matter Expert	Signature: <i>[Signature]</i>	Date: <i>[Signature]</i> <input type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Printed Name:		
Quality Assurance Manager	Signature: <i>[Signature]</i>	Date: <i>[Signature]</i> <input type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Printed Name:		
Chief Forensic Chemist	Signature: <i>[Signature]</i>	Date: 1/25/17 <input checked="" type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Printed Name: Robert Ramotowski		
Laboratory Director (Acting)	Signature: <i>Kelli Lewis</i>	Date: 6/16/17 <input checked="" type="checkbox"/> Approved <input type="checkbox"/> Revisions Required <input type="checkbox"/> Rejected
Printed Name: <i>Kelli Lewis</i>		
Comments: <i>Safety Officer - Approved Approved</i> <i>Brian Jones Brian A. Jones 2/3/17</i> <i>BC QAESB Kelli Lewis 2/3/17 approved</i> <i>w/ Revisions (Kelli Lewis)</i>		

FSD-056-F: FSD Method / Equipment Validation Form
Approved By: FSD Laboratory Director
All Printed Copies are Uncontrolled

Page 3 of 3

First Approved: Sep 2013
Revision #: NEW
Revision Effected: --

Conclusions

- Raw data is backed up on agency network.
- The PD working solution prepared using the USP grade was found to produce better results in 27% of the samples compared to 5% of the time for the ACS grade.
- The PD working solution prepared using the technical grade was found to produce better results in only 17% of the samples compared to 10% of the time for the ACS grade.
- There was a tendency at the lower end of the experience scale to determine that the two halves of a sample were equal (i.e., a grade of 0). The three IAI certified examiners were more likely to choose one side as being of better overall quality.



*U.S. Department of
Homeland Security*

United States
Secret Service

Conclusions

- Based on these results, a lower grade of silver nitrate (e.g., USP, technical) could be used in the physical developer working solution and that it would be more cost effective to forensic laboratories without sacrificing the overall quality of fingerprint development.



*U.S. Department of
Homeland Security*

United States
Secret Service

Contact Information

Robert Ramotowski
Chief Forensic Chemist
U.S. Secret Service
Forensic Services Division
950 H Street, NW Suite 4200
Washington, DC 20223
+1 202-406-6766 (tel)
+1 202-406-5603 (fax)

robert.ramotowski@uss.s.dhs.gov



*U.S. Department of
Homeland Security*

United States
Secret Service