Technical Procedure for Reagent Preparation

1.0 Purpose – To provide guidelines for preparing reagents used in casework in the Firearms Unit.

2.0 Scope – This procedure applies to all reagents used in the Firearms Unit.

3.0 Definitions – N/A

4.0 Equipment, Materials, and Reagents

- Acetic acid (99.7+ %, A.C.S Reagent)
- Birchwood Casey reagent (Brass Cartridge Case Cleaner)
- Cupric chloride
- Desensitized photographic paper
- Ethyl alcohol
- Ferric chloride
- Hydrochloric acid (37 % A.C.S. reagent)
- Hydrochloric acid (Certified A.C.S. *Plus*)
- Lead sheets (will be used but not consumed)
- Methanol
- Naphthol
- Nitric acid
- Sodium bitartrate
- Sodium hydroxide
- 10N Sodium hydroxide
- Sodium nitrite
- Sodium rhodizonate
- Sulfanilic acid
- Tartaric acid
- diH_2O (deionized water)
- Balance
- Beakers
- Cotton-tipped swabs
- Graduated cylinders
- Hot plate
- Magnetic stir rods
- Spatulas
- Stirring rods
- Weigh boats
- Fume hood
- Safety Data Sheets (SDS)

5.0 Procedure

5.1 15 % Acetic Acid Solution

5.1.1 Required Items

- Acetic acid (99.7+ %, A.C.S Reagent)
- diH_2O

5.1.2 Instructions for Preparation

5.1.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 1000 mL	For 4000 mL
Acetic acid	150 mL	600 mL
diH_2O	850 mL	3400 mL

5.1.2.2 Combine acetic acid with diH_2O.

5.1.3 Storage Conditions

5.1.3.1 Store at room temperature.

5.1.4 Expiration Date

5.1.4.1 Expires six (6) months after preparation.

5.2 Acidic Ferric Chloride Solution

5.2.1 Required Items

- Ferric chloride
- Hydrochloric acid (Certified A.C.S. *Plus*)
- diH_2O

5.2.2 Instructions for Preparation

5.2.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 125 mL	For 375 mL
Ferric chloride	25 g (385.81 gr)	75 g (1157.43 gr)
Hydrochloric acid	25 mL	75 mL
diH_2O	100 mL	300 mL

5.2.2.2 Combine ferric chloride with diH_2O. Then add hydrochloric acid and stir until the ferric chloride is no longer visible in the solution. The solution will appear as an orange color.

5.2.3 Storage Conditions

5.2.3.1 Store at room temperature in the top tray of the serial number kit or appropriate storage cabinet.

5.2.4 Expiration Date

5.2.4.1 Expires one (1) year after preparation.

5.3 Buffer Solution

5.3.1 Required Items

- Sodium bitartrate
- Tartaric acid
- diH_2O

5.3.2 Instructions for Preparation

5.3.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 1000 mL	For 1500 mL
Sodium bitartrate	293.0 gr	439.5 gr
Tartaric acid	231.0 gr	346.5 gr
diH_2O	1000 mL	1500 mL

5.3.2.2 Dissolve sodium bitartrate and tartaric acid in diH_2O. This usually requires both heat and agitation to complete in a reasonable amount of time.

5.3.3 Storage Conditions

5.3.3.1 Store at room temperature in an uncontaminated and sealed container.

5.3.4 Expiration Date

5.3.4.1 Expires one (1) month after preparation.

5.4 5% Cartridge Case Cleaning Solution

5.4.1 Required Items

- Birchwood Casey reagent (Brass Cartridge Case Cleaner)
- diH_2O

5.4.2 Instructions for Preparation

5.4.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 100 mL	For 500 mL
Birchwood Casey	5 mL	25 mL
diH_2O	95 mL	475 mL

5.4.2.2 Combine Birchwood Casey Cartridge Case Cleaner with diH_2O.

5.4.3 Storage Conditions

5.4.3.1 Store at room temperature.

5.4.4 Expiration Date

5.4.4.1 Expires one (1) year after preparation.

5.5 Davis Reagent Solution

5.5.1 Required Items

- Cupric chloride
- Hydrochloric acid (Certified A.C.S. *Plus*)
- diH_2O

5.5.2 Instructions for Preparation

5.5.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 100 mL	For 300 mL
Cupric chloride	77.16 gr	231.49 gr
Hydrochloric acid	50 mL	150 mL
diH_2O	50 mL	150 mL

5.5.2.2 Combine cupric chloride with deionized water. Then add hydrochloric acid and stir until the cupric chloride is no longer visible in the solution. The solution will appear as a dark green color.

5.5.3 Storage Conditions

5.5.3.1 Store at room temperature in the top tray of the serial number kit or appropriate storage cabinet.

5.5.4 Expiration Date

5.5.4.1 Expires one (1) year after preparation.

5.6 Ferric Chloride Solution

5.6.1 Required Items

- Ferric Chloride
- diH_2O

5.6.2 Instructions for Preparation

5.6.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 100 mL	For 300 mL
Ferric chloride	25 g (385.81 gr)	75 g (1157.43 gr)
diH_2O	100 mL	300 mL

5.6.2.2 Combine ferric chloride with diH_2O and stir until the ferric chloride is no longer visible in the solution. The solution will appear as an orange color.

5.6.3 Storage Conditions

5.6.3.1 Store at room temperature in the top tray of the serial number kit or appropriate storage cabinet.

5.6.4 Expiration Date

5.6.4.1 Expires one (1) year after preparation.

5.7 Fry's Reagent

5.7.1 Required Items

- Cupric chloride
- Hydrochloric acid (37 % A.C.S. reagent)

5.7.2 Instructions for Preparation

5.7.2.1 This solution may be mixed in any amount that maintains the original ratio.

Cupric chloride	enough for super-saturation
HCl	100 mL -200 mL

5.7.2.2 Dissolve cupric chloride in hydrochloric acid until the solution is super-saturation. Super-saturation occurs when cupric chloride will no longer go into solution. The saturated solution will appear as a very dark green color.

5.7.3 Storage Conditions

5.7.3.1 Store at room temperature in the top tray of the serial number kit or appropriate storage cabinet.

5.7.4 Expiration Date

5.7.4.1 Expires one (1) year after preparation.

5.8 Greiss Paper Solution

5.8.1 Required Items

- Sulfanilic acid
- diH_2O
- Naphthol
- Methanol

5.8.2 Instructions for Preparation

5.8.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 1000 mL	For 2500 mL	For 4000 mL
Sulfanilic acid	38.5 gr	96.25 gr	154.0 gr
diH_2O	500 mL	1250 mL	2000 mL
Naphthol	21.5 gr	53.75 gr	86.0 gr
Methanol	500 mL	1250 mL	2000 mL

5.8.2.2 Dissolve sulfanilic acid in diH_2O. Dissolve naphthol in methanol. Combine equal amounts of the sulfanilic acid and naphthol solutions.

5.8.3 Storage Conditions

5.8.3.1 Store at room temperature.

5.8.4 Expiration Date

5.8.4.1 Expires one (1) year after preparation.

5.9 Greiss Paper

5.9.1 Required Items

- Griess Paper solution, 100 mL – 200 mL
- Desensitized photographic paper (Note: filter paper may be processed in the same manner for use in the Modified Griess Test.)

5.9.2 Instructions for Preparation

5.9.2.1 Place Greiss paper solution in a non-reactive tray.

5.9.2.2 Briefly dip precut sheets of desensitized photographic paper into tray. Simply submerge each sheet and remove.

5.9.2.3 Hang sheets to dry and place the remaining Griess paper solution into an uncontaminated and sealed storage container.

5.9.2.4 Once the Greiss paper has dried, test a piece for sensitivity to nitrite compounds by saturating the tip of an untreated swab (control) with 15 % acetic acid solution and streaking the paper. Then saturate the tip of a nitrite swab with 15 % acetic acid solution and streak the same piece of Greiss paper in a different location. A positive test result occurs when an orange color appears on the paper where the nitrite swab was streaked. The streak made by the control should not turn orange.

5.9.3 Storage Conditions

5.9.3.1 Store at room temperature.

5.9.4 Expiration Date

5.9.4.1 None (made upon need).

5.10 5% Hydrochloric Acid Solution

5.10.1 Required Items

- Hydrochloric Acid (37% A.C.S. reagent)
- diH_2O

5.10.2 Instructions for Preparation

5.10.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 1500 mL	For 2000 mL	For 4000 mL
Hydrochloric acid	75 mL	100 mL	200 mL
diH_2O	1425 mL	1900 mL	3800 mL

5.10.2.2 Combine hydrochloric acid with diH_2O.

5.10.3 Storage Conditions

5.10.3.1 Store at room temperature.

5.10.4 Expiration Date

5.10.4.1 Expires six (6) months after preparation.

5.11 Lead Swabs

5.11.1 Required Items

- Lead sheets (will be used but not consumed)
- Cotton-tipped swabs, 100

5.11.2 Instructions for Preparation

5.11.2.1 Wipe the cotton tip of the swabs on the surface of the lead sheets, transferring lead to the cotton swabs.

5.11.2.2 Test the lead swabs by performing the Sodium Rhodizonate Test for lead residue. An untreated swab (control) should be tested as well. A positive result indicating the presence of lead occurs when the lead swab turns a blue-violet color following the spraying of 5 % hydrochloric acid solution. The control should not turn blue-violet.

5.11.3 Storage Conditions

5.11.3.1 Store at room temperature.

5.11.4 Expiration Date

5.11.4.1 Lead swabs do not expire.

5.12 25% Nitric Acid Solution

5.12.1 Required Items

- Nitric acid
- diH_2O

5.12.2 Instructions for Preparation

5.12.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 300 mL
Nitric acid	75 mL
diH_2O	225 mL

5.12.2.2 Add nitric acid to diH_2O and mix.

5.12.3 Storage Conditions

5.12.3.1 Store at room temperature in the top tray of the serial number kit or appropriate storage cabinet.

5.12.4 Expiration Date

5.12.4.1 Expires one (1) year after preparation.

5.13 Nitrite Swabs

5.13.1 Required Items

- Sodium nitrite
- diH_2O
- Cotton-tipped swabs, 100

5.13.2 Instructions for Preparation

5.13.2.1 Dissolve sodium nitrite in water.

5.13.2.2 Soak the cotton-tipped ends of the swabs in the solution.

5.13.2.3 Dry the swabs by placing them upside down (cotton tip up) in a beaker.

5.13.2.4 Test the nitrite swabs by saturating the tip of an untreated swab (control) with 15 % acetic acid solution and streaking a piece of Greiss paper. Then saturate a freshly prepared nitrite swab with 15 % acetic acid solution and streak the same piece of Greiss paper in a different location. A positive test result occurs when an orange

streak is produced by the nitrite swab. The streak made by the control swab should be clear.

5.13.3 Storage Conditions

5.13.3.1 Store at room temperature.

5.13.4 Expiration Date

5.13.4.1 Nitrite swabs do not expire.

5.14 10% Sodium Hydroxide Solution

5.14.1 Required Items

- Sodium hydroxide
- diH_2O

5.14.2 Instructions for Preparation

5.14.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 90 mL	For 270 mL
Sodium hydroxide	10 g (154.32 gr)	30 g (462.97 gr)
diH_2O	90 mL	270 mL

5.14.2.2 Dissolve sodium hydroxide in diH_2O until the pellets are no longer visible.

5.14.3 Storage Conditions

5.14.3.1 Store at room temperature in the top tray of the serial number kit or appropriate storage cabinet.

5.14.4 Expiration Date

5.14.4.1 Expires one (1) year after preparation.

5.15 2N Sodium Hydroxide Solution

5.15.1 Required Items

- 10N sodium hydroxide or sodium hydroxide
- diH_2O

5.15.2 Instructions for Preparation (Method 1)

5.15.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 100 mL	For 500 mL
10N sodium hydroxide	20 mL	100 mL

diH_2O	80 mL	400 mL

5.15.2.2 Combine 10N sodium hydroxide with diH_2O. The volume prepared should be only the necessary volume for appropriate waste disposal.

5.15.3 Instructions for Preparation (Method 2)

5.15.3.1 This solution may be mixed in any amount that maintains the original ratio.

	For 100 mL
Sodium hydroxide	123.46 gr
diH_2O	100 mL

5.15.3.2 Dissolve sodium hydroxide in diH_2O until the pellets are no longer visible. The volume prepared should be only the necessary volume for appropriate waste disposal.

5.15.4 Storage Conditions

5.15.4.1 N/A – Only enough solution is made for immediate use.

5.15.5 Expiration Date

5.15.5.1 N/A – Only enough solution is made for immediate use.

5.16 Sodium Rhodizonate

5.16.1 Required Items

- Sodium rhodizonate
- diH2O

5.16.2 Instructions for Preparation

5.16.2.1 This solution may be mixed in any amount that maintains the original ratio.

Sodium rhodizonate	enough for saturation
diH2O	10 mL – 20 mL

5.16.2.2 Combine a small amount of sodium rhodizonate with diH_2O until solution is saturated. The solution is saturated if slight sediment is noted on the bottom of the beaker after stirring with a clean glass stirring rod. The saturated solution will appear approximately the color of strong tea.

5.16.3 Storage Conditions

5.16.3.1 N/A – Only enough solution is made for immediate use.

5.16.4 Expiration Date

5.16.4.1 N/A – Only enough solution is made for immediate use.

5.17 Turner's Reagent

5.17.1 Required Items

- Cupric chloride
- Hydrochloric acid (Certified A.C.S. *Plus*)
- Ethyl alcohol
- diH_2O

5.17.2 Instructions for Preparation

5.17.2.1 This solution may be mixed in any amount that maintains the original ratio.

	For 100 mL	For 300 mL
Cupric chloride	38.58 gr	115.74 gr
Hydrochloric acid	40 mL	120 mL
Ethyl alcohol	25 mL	75 mL
diH_2O	30 mL	90 mL

5.17.2.2 Combine cupric chloride with diH_2O. Then add hydrochloric acid and ethyl alcohol. Stir until cupric chloride is no longer visible in the solution. The solution will appear as a light green color.

5.17.3 Storage Conditions

5.17.3.1 Store at room temperature in the top tray of the serial number kit or appropriate storage cabinet.

5.17.4 Expiration Date

5.17.4.1 Expires one (1) year after preparation.

5.18 Standards and Controls

5.18.1 For Greiss paper testing, see 5.9.2.4.

5.18.2 For lead swab control testing, see 5.11.2.2.

5.18.3 For nitrite swab control testing, see 5.13.2.4.

5.19 Calibration – N/A

5.20 Maintenance – N/A

5.21 Sampling – N/A

5.22 Calculations – N/A

5.23 Uncertainty of Measurement – N/A

6.0 Limitations – N/A

7.0 Safety – N/A

8.0 References – N/A

9.0 Records

- Reagent Prep Logs

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original Document
02/15/2013	2	Removed Raleigh from the header; 4.0 – added fume hood and MSDS; added "or appropriate storage cabinet" to 5.2.3.1, 5.6.3.1, 5.7.3.1, 5.12.3.1, 5.14.3.1, and 5.17.3.1; created new 5.5; added "or sodium hydroxide" to 5.15.1 ; 5.15.2 – added language in parentheses; created new 5.15.3, 5.15.3.1, and 5.15.3.2
11/15/2013	3	Added issuing authority to header
09/05/2014	4	**Header and various subsections** – corrected to reflect organizational change; grammar
09/22/2017	5	Updated header information 4.0-MSDS to SDS 5.18.1, 5.18.2, 5.18.3 – fix referenced numbers