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## Technical Procedure for Reagent Preparation

**1.0 Purpose** – To provide guidelines for preparing reagents used in casework in the Firearm and Tool Mark Section.

**2.0 Scope** – This procedure applies to all reagents used in the Firearm and Tool Mark Section.

**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<sub>2</sub>O (deionized water)
- Balance
- Beakers
- Cotton-tipped swabs
- Graduated cylinders
- Hot plate
- Magnetic stir rods
- Spatulas
- Stirring rods
- Weigh boats
- Fume hood
- Material Safety Data Sheets (MSDS)

### **5.0 Procedure**

#### **5.1 15 % Acetic Acid Solution**

##### **5.1.1 Required Items**

- Acetic acid (99.7+%, A.C.S Reagent)
- diH<sub>2</sub>O

### 5.1.2 Instructions for Preparation

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

	<u>For 1000 mL</u>	<u>For 4000 mL</u>
Acetic acid	150 mL	600 mL
diH <sub>2</sub> O	850 mL	3400 mL

5.1.2.2 Combine acetic acid with diH<sub>2</sub>O.

### 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<sub>2</sub>O

### 5.2.2 Instructions for Preparation

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

	<u>For 125 mL</u>	<u>For 375 mL</u>
Ferric chloride	25 g (385.81 gr)	75 g (1157.43 gr)
Hydrochloric acid	25 mL	75 mL
diH <sub>2</sub> O	100 mL	300 mL

5.2.2.2 Combine ferric chloride with diH<sub>2</sub>O. 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<sub>2</sub>O

#### 5.3.2 Instructions for Preparation

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

	<u>For 1000 mL</u>	<u>For 1500 mL</u>
Sodium bitartrate	293.0 gr	439.5 gr
Tartaric acid	231.0 gr	346.5 gr
diH <sub>2</sub> O	1000 mL	1500 mL

5.3.2.2 Dissolve sodium bitartrate and tartaric acid in diH<sub>2</sub>O. 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<sub>2</sub>O

#### 5.4.2 Instructions for Preparation

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

	<u>For 100 mL</u>	<u>For 500 mL</u>
Birchwood Casey	5 mL	25 mL
diH <sub>2</sub> O	95 mL	475 mL

5.4.2.2 Combine Birchwood Casey Cartridge Case Cleaner with diH<sub>2</sub>O.

#### 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<sub>2</sub>O

**5.5.2** Instructions for Preparation

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

	<u>For 100 mL</u>	<u>For 300 mL</u>
Cupric chloride	77.16 gr	231.49 gr
Hydrochloric acid	50 mL	150 mL
diH <sub>2</sub> O	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<sub>2</sub>O

**5.6.2** Instructions for Preparation

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

	<u>For 100 mL</u>	<u>For 300 mL</u>
Ferric chloride	25 g (385.81 gr)	75 g (1157.43 gr)
diH <sub>2</sub> O	100 mL	300 mL

**5.6.2.2** Combine ferric chloride with diH<sub>2</sub>O 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<sub>2</sub>O
- 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.

	<u>For 1000 mL</u>	<u>For 2500 mL</u>	<u>For 4000 mL</u>
Sulfanilic acid	38.5 gr	96.25 gr	154.0 gr
diH <sub>2</sub> O	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<sub>2</sub>O. 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<sub>2</sub>O

### **5.10.2** Instructions for Preparation

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

	<u>For 1500 mL</u>	<u>For 2000 mL</u>	<u>For 4000 mL</u>
Hydrochloric acid	75 mL	100 mL	200 mL
diH <sub>2</sub> O	1425 mL	1900 mL	3800 mL

**5.10.2.2** Combine hydrochloric acid with diH<sub>2</sub>O.

### **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<sub>2</sub>O

#### 5.12.2 Instructions for Preparation

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

	<u>For 300 mL</u>
Nitric acid	75 mL
diH <sub>2</sub> O	225 mL

5.12.2.2 Add nitric acid to diH<sub>2</sub>O 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<sub>2</sub>O
- 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<sub>2</sub>O

### 5.14.2 Instructions for Preparation

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

	<u>For 90 mL</u>	<u>For 270 mL</u>
Sodium hydroxide	10 g (154.32 gr)	30 g (462.97 gr)
diH <sub>2</sub> O	90 mL	270 mL

5.14.2.2 Dissolve sodium hydroxide in diH<sub>2</sub>O 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

- 10 N sodium hydroxide or sodium hydroxide
- diH<sub>2</sub>O

### 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.

	<u>For 100 mL</u>	<u>For 500 mL</u>
10 N sodium hydroxide	20 mL	100 mL

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diH <sub>2</sub> O	80 mL	400 mL
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**5.15.2.2** Combine 10 N sodium hydroxide with diH<sub>2</sub>O. 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.

	<u>For 100 mL</u>
Sodium hydroxide	123.46 gr
diH <sub>2</sub> O	100 mL

**5.15.3.2** Dissolve sodium hydroxide in diH<sub>2</sub>O 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
- diH<sub>2</sub>O

**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
diH <sub>2</sub> O	10 mL – 20 mL

**5.16.2.2** Combine a small amount of sodium rhodizonate with diH<sub>2</sub>O until solution is saturated. The solution is saturated if a 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<sub>2</sub>O

### **5.17.2 Instructions for Preparation**

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

	<u>For 100 mL</u>	<u>For 300 mL</u>
Cupric chloride	38.58 gr	115.74 gr
Hydrochloric acid	40 mL	120 mL
Ethyl alcohol	25 mL	75 mL
diH <sub>2</sub> O	30 mL	90 mL

**5.17.2.2** Combine cupric chloride with diH<sub>2</sub>O. 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 [5.8.2.4](#).

**5.18.2** For lead swab control testing, see [5.9.2.2](#).

**5.18.3** For nitrite swab control testing, see [5.12.2.4](#).

**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**

<b>Revision History</b>		
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