1. REAGENT PREPARATION AND USE

A. Refer to TR-SOP-12 for general guidelines of chemical comparison and examination.

B. The date of preparation and the lot numbers of chemicals used in the preparation of the reagent will be documented in a readily accessible logbook. A lot number (normally the date of preparation), and expiration date will be assigned to the prepared reagent and marked on the container.

C. Prepared reagents may be obtained from other sections of the Laboratory Services Bureau for use in the Trace section. The lot number, expiration date, positive and negative controls of these reagents will be documented in the case notes prior to use.

D. Expiration dates of prepared reagents will be 1 year from the time of preparation unless specifically noted.

E. Preparation and use are as follows for commonly used reagents:

(1) Copper Detection

(a) Preparation of 28% AMMONIA HYDROXIDE SOLUTION

- Place 143 milliliters of concentrated NH4OH in 357 milliliters of distilled water.

(b) Preparation of DITHIOOXAMIDE SOLUTION

- Dissolve 0.2 grams of Dithiooxamide in 100 milliliters of ethyl alcohol.

(c) Moisten an area of filter paper of adequate size to sample the area of interest with the 28% ammonium hydroxide solution. Firmly press the moistened filter paper on the sample for a minimum of 15 seconds.

(d) Remove the filter paper and add the Dithiooxamide solution dropwise to cover the sampled area.

(e) Note the development of color and compare to the result that is obtained with a standard. The development of a comparable color indicates the presence of copper.

(2) Lead Detection

(a) Preparation of 15% ACETIC ACID SOLUTION

- Place 75 milliliters of concentrated Glacial Acetic Acid in 425 milliliters of distilled water.

(b) Preparation of 5% HYDROCHLORIC ACID SOLUTION

- Place 25 milliliters of concentrated Hydrochloric Acid in 475 milliliters of distilled water.

(c) Preparation of 0.1% (approximate) SODIUM RHODIZONATE SOLUTION

- Place a sufficient amount of Sodium Rhodizonic acid (normally the tip of a spatula) into a dropper vial filled with approximately 5-10 milliliters of distilled water to produce a dark brown color, similar to tea in appearance.

- Prepare at time of testing and discard upon completion of testing.

(d) Moisten an area of filter paper of adequate size to sample the area of interest with the 15% Acetic acid solution. Firmly press the moistened filter paper on the sample for a minimum of 15 seconds.

(e) Remove the filter paper and add the Sodium Rhodizonate solution drop wise to cover the sampled area.

(f) Note the development of color and compare to the result that is obtained with a standard. The development of a comparable color indicates the presence of lead.

(g) Add the 5% Hydrochloric acid drop wise to the area of color obtained with the Sodium Rhodizonate solution.

(h) Note the development of color and compare to the result that is obtained with a standard. The development of a comparable color is an identification of the presence of lead.

(3) Nitrate and Nitrite Ion Detection

(a) Preparation of DIPHENYLBENZIDINE SOLUTION

- Place 1 milligram of Diphenylbenzidine in 10 milliliters of concentrated Sulfuric Acid. Store in a brown dropper bottle.

(b) Add the Diphenylbenzidine solution drop wise to a representative portion of the sample in an appropriate container such as a spot plate.

(c) Note the development of color and compare to the results obtained with a standard. The development of a comparable color indicates the presence of both the nitrate and nitrite ion.

(4) Nitrite or Nitrate Ion Detection

(a) Preparation of GRIESS' REAGENT

- Dissolve 0.25 grams Sulfanilic acid and 0.1 g l-Naphthylamine in 100 milliliters 1:1 (50%) aqueous Acetic Acid.

(b) Add the Griess' Reagent dropwise to a representative portion of the sample in an appropriate container such as a spot plate.

(c) Note the development of color and compare to the results obtained with a standard. This is a detection of the nitrite ion.

(d) If no color develops add a small portion of powdered zinc to the solution.

(e) Note the development of color and compare to the results obtained with a standard. The development of a comparable color indicates the presence of the nitrate ion.

(5) Ammonium Ion Detection

(a) Preparation of NESSLER'S REAGENT

- Dissolve 5 grams HgI2 and 2.5 grams KI in 25 milliliters of distilled water. Dissolve 10 grams KOH in 25 milliliters distilled water and add to the above solution.

(b) Add the Nessler's Reagent dropwise to a representative portion of the sample in an appropriate container such as a spot plate.

(c) Note the development of color and compare to the results obtained with a standard. The development of a comparable color indicates the presence of the ammonium ion.