

Name of Procedure:

Toxicology
Lead Screen

Suggested Uses:

This procedure will screen for the presence of lead to approximately 1 ppm in a liquid sample. This test is commonly used to test liquor samples for the presence of high levels of lead. The test is based on dithizone color reaction.

Items Used to Perform Procedure:

Nitric acid solution, 0.166M
Lead stock solution, 0.1%
Lead reference solutions
Dithizone stock solution, 5 mg%
Dithizone working solution, 1.25 mg%
Cyanide solution, 5% (0.768M)
Ammonium hydroxide, conc.
Test tubes, disposable 13x100 mm
Vortexer
Volumetric flasks 100 mL, 1 L, and 2 L

Reagent Preparation Procedure:

Note: These are guides for preparing reagents: It is acceptable to modify the amounts and volumes as long as the reagents made are the same concentration as listed.

1. Nitric acid solution, 0.166M
 - a. Dilute 20 mL of concentrated nitric acid to 2 liters, with deionized water.
2. Lead stock solution, 0.1%
 - a. Dissolve 1.5984 grams of lead nitrate ($\text{Pb}(\text{NO}_3)_2$) in the nitric acid solution, and bring the volume to a total of 1 L with the nitric acid solution.
3. Lead reference solutions (0.5, 1, 5, 10, and 25 ppm)
 - a. Dispense 50, 100, 500, 1000, and 2500 microliters of the 0.1% lead stock solution into four 100 mL volumetric flasks.

Drug Chemistry Section
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- b. Bring the volume of each volumetric flask to the 100 mL mark using nitric acid solution, 0.166M.
- 4. Dithizone stock solution, 5 mg%
 - a. dissolve 5 mg of dithizone (diphenylthiocarbazone) in 100 mL of high purity chloroform.
 - b. Label bottle with a 90-day expiration date.
- 5. Dithizone working solution, 1.25 mg%
 - a. 1. Dilute 25 mL of the Dithizone stock solution, 5 mg%, to a total volume of 100mL with chloroform. **or**
2. Dissolve 1.25mg of dithizone in 100mL of chloroform.
 - b. Label bottle with a 30-day expiration date.
- 6. Cyanide solution, 5%
Dissolve 2.0 grams of sodium cyanide or potassium cyanide in 40mL of deionized water.

Application of Procedure on Evidence:

- 1. Comparison / Control preparation:
As a point of comparison and to test for contamination of reagents, prepare a sample of deionized water and one or more appropriate lead reference solutions using the Sample analysis procedure.
- 2. Sample analysis:
 - a. Transfer 2 mL of protein free specimen to a 13x100 mm test tube.
 - b. Add 0.250 mL of ammonium hydroxide to the test tube.
 - c. Check the pH, which must be between 10-12.
 - d. Vortex.
 - e. Add 4-5 drops of 5% cyanide solution.
 - f. Vortex.
 - g. Add 1mL of the 1.25 mg% dithizone working solution.
 - h. Vortex for about 20 seconds.
 - i. Check bottom layer (chloroform) for a red to violet color.
 - j. If a pink-red color is present in the chloroform layer the test is positive for lead. Comparison to the lead reference solutions can give an approximate value of the lead concentration.

3. Positive results:
 - a. Any positive results must be confirmed using a more sensitive and specific test, such as atomic absorption, before identifying the presence of lead.
 - b. This test should be used as a screening test only.

Comments:

1. A red color in the chloroform layer, could also be indicative of the presence of thallium, bismuth, and/or stannous ions.
2. Most samples will have a small amount of lead (less than 0.5 ppm). There is no definitive answer as to what is an acceptable amount of lead, which is a cumulative poison. The World Health Organization has set the Acceptable Daily Intake for lead at 0.007 mg/kg body weight. (Seiler)

Safety Concerns:

When working with acids and bases here are a few rules to remember:

- a. Always add acid to water, not water to acid.
- b. Always wear gloves, protective clothing, and eye protection.
- c. Be careful when working with acids and bases together.
- d. Sodium and potassium cyanide are extremely toxic.

Literature References:

Sunshine, Irving, **Methodology for Analytical Toxicology**, CRC Press, 1955, pp. 204-209.

Kaye, Sidney, **Handbook of Emergency Toxicology**, Thomas, Charles C., (publisher), 1954, pp. 202-208.

Seiler, Sigel, Sigel, **Handbook on Toxicity of Inorganic Compounds**, 1988.