NATIONAL PARK SERVICE
DEPARTMENT OF THE INTERIOR

Conserve O Gram

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Testing For Chlorides With Silver Nitrate

The deterioration of metals, particularly iron, is greatly accelerated when the metal is contaminated with chloride ions. Chloride ions are found in table salt, in sea water, on skin, and in the soil. It is a problem with buried objects, objects recovered from the sea, and metals handled with bare, sweaty fingers. Should it become necessary for park staff to test these objects, the following procedures can be attempted under the direction of a conservator.

In reducing the chloride levels in iron by electrolysis or a passive alkaline soak (see *Conserve O Gram* 6/2), the progress of the treatment can be monitored by testing the rinse water for the presence of chloride ions flushed from the object. The simplest method for detecting chlorides uses silver nitrate which reacts with the chlorides to form a cloudy white precipitate.

Materials Needed for the Test

- Distilled or deionized water.
- Test tube, rinsed in distilled water.
- Dilute (5%) nitric acid, in a dropper bottle.
- Silver nitrate test solution, in a dropper bottle.
- Black or dark background with a strong side light.

To Make Test Solution

Dissolve 1.7 grams (g) of silver nitrate crystals into 98 milliliters (ml) of distilled water. Store the solution in a dropper bottle in a dim or dark place, because it is somewhat light sensitive.

Procedure

1. Place a sample of the alkaline bath water (10 to 30 ml) in the clean test tube.

2. Add one or two drops of dilute nitric acid. Shake to mix. If the solution fizzes, continue to add acid until the fizz stops.

3. Hold the test tube up to a strong side light with a dark area behind the test tube. Add two drops of the test solution. Observe the sample closely.

4. The formation of a white precipitate (i.e., a cloudiness) indicates the presence of chlorides. If the cloudiness is so weak as to raise doubts as to its presence, compare it with an *untested* sample of the rinse water.

5. Thoroughly rinse the test tube with distilled or deionized water to prepare for the next sample.

To Test for Chlorides in Objects Not Yet in Treatment

1. Prepare the object by cleaning off as much adhering soil and loose encrustation as practical (e.g., wash, brush, chip).

2. Place the object in just enough distilled water to barely cover it.

3. Let the object soak for one day. This will allow time for the chlorides to diffuse into the water. If the contamination is very

great, a detectable amount of chlorides can diffuse in minutes.

4. Take a sample of the rinse water, 10 to 30 ml, in a clean test tube. If the water is too rusty or cloudy, filter it through clean filter papers.

5. Test as previously specified.

Interpreting Test Results

1. If the chloride concentrations are below 100 parts per million (ppm), the degree of cloudiness is roughly in proportion to the chloride level. Above 100 ppm, all the reactions look equally cloudy. Thus, 250 ppm looks as cloudy as 2500 ppm.

2. With careful technique (clean test tubes, good sidelight, accurately prepared silver nitrate test solution), concentrations as low as 1 ppm may be detected.

3. There will likely be some detectable chlorides in the chemicals used to make up the alkaline-soaking solutions, so test the solutions *before* the object is immersed, as well as after soaking. The raw water used to make up the solutions should also be tested to provide a base line.

4. To provide points of comparison, make up sample solutions with known concentrations of chlorides.

   a. Obtain a volumetric flask of one liter capacity, and a second of 100 ml capacity; four storage bottles, one liter each, of polyethylene or glass; a few grams of reagent grade sodium chloride; distilled or deionized water for rinsing and cleaning.

   b. Fill a clean one-liter volumetric flask almost to the mark with distilled water. Into this, dissolve 1.65 grams of reagent grade sodium chloride. Add more water until the mark is reached. This will be a 1000 ppm solution.

   c. To make a 100 ppm solution, fill a clean 100 ml volumetric flask with a 1000 ppm solution. Pour this into a clean one liter volumetric flask. Fill to the mark with distilled water. This will dilute the 1000 ppm solution to 100 ppm.

   d. To make a 10 ppm solution, dilute the 100 ppm solution as above.

   e. To make a 1 ppm solution, dilute the 10 ppm solution as above.

   f. Always rinse the flasks thoroughly in distilled water to avoid contamination of successive solutions.

5. To use the comparison solutions, pour a 10 to 30 ml quantity of each known concentration as prepared above (1 ppm, 10 ppm, 100 ppm, and 1000 ppm) into separate test tubes, at the same time as a rinse solution of unknown concentration is being tested. A visual comparison of the densities of the white precipitates in each solution should give a rough idea of between which two values the unknown solution appears to fall.

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