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A COMPARISON OF METHODS FOR RECYCLING SILVER IN LABORATORY WASTES

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ABSTRACT

The price of silver nitrate, now about $300 per 500 grams, suggests the worth of recycling silver from lab wastes if efficient methods can be developed for its conversion to silver nitrate. Two methods are described for recycling silver with 95% efficiency. The techniques yield silver nitrate of adequate purity for use as a quantitative analysis reagent.

INTRODUCTION

A primary use of silver nitrate is in the gravimetric determination of chloride by quantitative analysis students. Silver nitrate (reagent grade, 99.9+% pure) is selling for $139.50—$425.65 per 500 grams (Alfa Products, January, 1985; Aldrich, February, 1984; respectively). This price suggests the worth of recycling silver from lab wastes if efficient methods can be developed for its conversion to silver nitrate. Waste silver nitrate solutions from the student labs have been stockpiled for years, and last year we began saving the silver chloride prepared by students. The department of Chemistry and Physics has a policy of recycling chemical reagents and solvents whenever feasible to reduce the trouble and expense of waste disposal, but the high cost of silver nitrate was further impetus for devising a procedure for its reclamation. Two methods, both inexpensive and fast, are described for recycling silver nitrate with 95% efficiency. Both are reductions: the first with copper turnings, the second with sodium borohydride.

METHODS

As a preliminary experiment, the procedure of Thall was followed to convert silver chloride, in 6 M ammonium hydroxide solution, to silver using copper strips. The product was a mixture; it was suspected to be contaminated with unreacted silver chloride and copper metal. Analysis of the product using scanning electron microscopy showed the presence of needles which were identical in appearance to the needles in an authentic silver chloride sample. Thall's note lacked procedural details but it is possible that the quantity of ammonium hydroxide used was insufficient to dissolve all the silver chloride in the sample. The modified procedure below avoids contaminated samples and results in greater than 95% recovery of silver. From ammoniacal silver solutions, highly explosive compounds may precipitate; such solutions should never be stored. This procedure should be carried out with caution and in the suggested quantities, since diamminesilver ion, $Ag(NH_3)_2^+$, is reported to be explosive.

> Add 1.0 to 20.0 g of pulverized silver chloride to 500 mL deionized water. Stir the mixture and filter it. Add to the washed silver chloride, 85 mL of conc. ammonium hydroxide solution (15 M) for each gram of silver chloride, and stir until the silver chloride dissolves. Add 5 g of heavy copper turnings or copper strips for each gram of silver chloride dissolved. Allow to stand overnight. Remove the unreacted pieces of copper with tweezers; filter, wash, and oven-dry the silver metal (yield: 0.75 g silver/g silver chloride, 99.7%). The unreacted copper can be reused.

A second method for reclaiming silver was a treatment of silver nitrate solutions with the mild reducing agent, sodium borohydride. When sodium borohydride is added to silver nitrate solutions there is a rapid evolution of hydrogen gas as the silver ions are reduced to silver metal, so these reactions were conducted in a well ventilated hood.

Hydrogen presents both combustion-explosion and fire hazards. It should be generated only in areas free of ignition sources. Ventilation is the most effective way to prevent formation of an explosive mixture of air and hydrogen; therefore, an exhaust hood must be used whenever mixtures generating appreciable quantities of hydrogen are transferred, allowed to stand, heated, or handled in any other way.

The procedure of Hohnstedt, et. al., was followed in the initial experiments to reclaim silver from silver nitrate solutions using sodium borohydride as the reducing agent. Later the concentration of sodium borohydride was increased to speed the reactions.

> To 4.25 g silver nitrate in 25.0 mL of d.i. water (1 M silver nitrate) was added dropwise with stirring, 50 mL of 1 M sodium borohydride solution. After two hours, the metal precipitate was collected in a sintered glass crucible, washed with d.i. water, then with acetone, and oven dried (yield: 2:66 g silver, 98.6%).

For waste silver nitrate solutions, no effort was made to determine the concentration of silver nitrate. These solutions (250 mL portions) were adjusted to pH 7 with 1 M NaOH, and then treated with 1% sodium borohydride solution, added dropwise with stirring. After the evolution of hydrogen gas was complete, the metallic silver was collected in a filter crucible, washed and oven-dried as before.

Silver metal was stored until it was convenient to convert it to silver nitrate according to the following procedure.

To 4.0 g of silver metal, was added 50 mL of 6 M nitric acid. After the silver dissolved, the solution was filtered through a sintered glass crucible. The volume of aqueous silver nitrate was reduced to about 10 mL on a hotplate. The residue was oven dried (yield: 6.19 g, 98.3%).

Nitrogen dioxide gas forms slowly as silver is oxidized to silver nitrate by nitric acid.

$$Ag + 2\ HNO_3 \rightarrow AgNO_3 + NO_2 + H_2O$$

Nitrogen dioxide is a reddish brown, deadly poisonous gas, and even short exposures to it at levels greater than 200 ppm may cause lung inflamation, edema, and death. Inhalation must be avoided by performing the reaction above under an exhaust hood.

DISCUSSION

The methods have been used successfully to reclaim silver from the chloride and for recycling old silver nitrate solutions to silver nitrate of adequate purity for use as a quantitative reagent.

Important factors in deciding to reclaim silver from laboratory wastes are the relative costs of the reducing agents and silver nitrate. These costs vary. Copper is about $30 per 500 grams and sodium borohydride is about $25.00 for 25 grams (reagent grade, Fisher, 1984). Scrap copper, which costs far less than reagent grade, can be used for reduction of the silver diammine complex. As a reducing agent, sodium borohydride is fast and efficient. One gram will reduce 18.0 g of silver nitrate to 11.4 g of silver metal. There were several opened, pound cans of sodium borohydride in two organic synthesis labs when we began this project. Organic chemists usually will not risk exposure of hard-won synthetic intermediates to reagents of unknown purity, so it was easy to obtain the sodium borohydride "free." Its consumption in reclaiming silver therefore solved two disposal problems. Use of sodium borohydride as the reducing agent, instead of copper, saves time. Students did all this work without pay. If labor costs have to be included, the cost of the silver nitrate, recycled using either technique, might be higher than $300 per pound.

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DENSITY ESTIMATION OF A RACCOON POPULATION IN WESTERN TENNESSEE

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ABSTRACT

Density was assessed for a population of raccoons (*Procyon lotor*) during March of 1982 using mark-recapture techniques. The study was conducted on an upland hardwood forest site at Land Between The Lakes in Stewart County, Tennessee. Twelve raccoons were captured; seven were recaptured one or more times. Density was one per 34.3 ha.

INTRODUCTION

Various investigators have estimated the population density of raccoons (*Procyon lotor*) in a variety of habitats and regions of North America. Lotze and Anderson (1979) summarized much of the available literature. Density estimates for raccoons are difficult to derive. This is partly due to the physical difficulties in working with a species having a large home range and to the fact that in most cases the number of animals included in a sample will be small. However, difficulties in estimating abundance does not exclude the need for such information. Management programs are difficult to construct and implement without knowledge relating to population density.

Minser and Pelton (1982) summarized raccoon density estimates for areas in Tennessee. With the exception of Moore and Kennedy (in press), no estimates are available from western Tennessee. The purpose of this study was to

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