Literature Cited

(1) Kagel, R. A.; Farwell, S. O. *J. Chem. Educ.* 1983, *60*, 163.
(2) Haddad, P.; Hutchins, S.; Tuffy, M. *J. Chem. Educ.* 1983, *60*, 166.
(3) Ondrus, M. G.; Wenzel, J.; Zimmerman, G. L. *J. Chem. Educ.* 1983, *60*, 776.
(4) Beaver, R. W.; Bunch, J. E.; and Jones, L. A.; *J. Chem. Educ.* 1983, *60*, 1000.
(5) Cantwell, F. F.; Brown, D. W. *J. Chem. Educ.* 1981, 58, 820.
(6) Jezorek, J. R.; Freiser, H. *Anal. Chem.* 1979, *51*, 366.
(7) Risner, C., Master's Thesis, University of North Carolina at Greensboro, May, 1984.
(8) Fritz, J. S.; Story, J. N. *Anal. Chem.* 1974, *46*, 825.
(9) Jezorek, J. R.; Freiser, H. *Anal. Chem.* 1979, *51*, 373.
(10) Elchuk, S.; Cassidy, R. M. *Anal. Chem.* 1979, *51*, 1434.
(11) Shibata, S. In "Chelates in Analytical Chemistry"; Flaschka, H., Barnard, A. J., Eds.; Marcel Dekker: New York, 1972; Vol 4, pp 116–165.
(12) Habig, R. L.; Pardue, H. L.; Worthington, J. B. *Anal. Chem.* 1967, *39*, 600.
(13) Crumbliss, A. L.; McCabe, M. E.; Dilts, J. A.; Herman, H. B. *J. Chem. Educ.* 1976, 53, 528.
(14) Ruzicka, J. *Anal. Chem.* 1983, 55, 1040A.
(15) Betteridge, D. *Anal. Chem.* 1978, *50*, 832A.
(16) Nikelly, J. G.; Ventura, D. A. *Anal. Chem.* 1979, *51*, 1585.
(17) Snyder, L. R.; Kirkland, J. J. "Introduction to Modern Liquid Chromatography", 2nd ed.; Wiley-Interscience: New York, 1979; p 117.

Production or Recovery of Silver for Laboratory Use

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One factor of major consideration of a laboratory with a limited budget is the cost of expensive chemicals. This includes silver nitrate, which is currently retail priced at around \$24–38 for 25 g and \$75–140 for 100 g. This paper is involved with a method by which teachers can either produce their own silver of "technical" quality or recycle silver chloride residues to recover the silver for similar reuse.

Perlman et al.[2] have described a method by which the silver diammine complex ion (made by dissolving silver chloride into ammonia) is reduced by ascorbic acid. The relative quantities he described were approximately 9 g of ascorbic acid to 18 g of silver chloride to produce 13.7 g of silver. This silver chloride can come from either recycled laboratory residues or, as we are showing here, it can be precipitated from solutions made by dissolving silver-bearing scraps. These methods of recovery are described here.

Recovery of Silver

Silver plate on heavy items (silverware, silver plated electronic connectors, etc.) can be stripped by holding them with steel crucible tongs or suspending them on either steel or stainless steel wire and dipping them into a beaker with warm concentrated nitric acid. The pieces are left only until most to all of the silver plate is dissolved off, then the piece is removed and rinsed with water to remove any adhering solution. This stripping solution is eventually diluted with at least five volumes of water then the silver is precipitated by adding an excess of either dilute hydrochloric acid or calcium chloride solution. The precipitate is separated from the supernatant and washed several times with water[3] to remove any adsorbed solutes containing other metals such as copper, nickel, zinc, etc. This precipitate should be stored wet for future reductions as the wet silver chloride dissolves into ammonia much better than the dry crystals.

High content items such as silver electrical contacts, sterling silver and jewelry, silver solder scrap, etc. are dissolved in concentrated nitric acid and the resulting solution is handled as described above to separate the silver chloride.

Reduction of Silver Chloride

The wet silver chloride is dissolved by stirring it with concentrated ammonia ("ammonium hydroxide") and using enough to dissolve the precipitate. At this point the solution may contain undissolved material (as hydroxides) which should be separated by decantation and filtration as appropriate. The resulting clear solution will likely be blue due to the copper ammine complex ion and color is an indication of purity at this point.

To the solution add an excess of ascorbic acid[4] as approximately 1 molar in water. When there is any question of excess, more should be added to insure a completeness of the reaction.

The silver precipitates as a fine gray powder which will slowly settle out of the reaction medium. Other metal ions do not appear to be affected by the ascorbic acid. Most of the supernatant can be decanted off for disposal and the silver powder is then rinsed[3] with water to remove any adhering metal salts. After final decantation, the silver is washed into a filter paper cone with acetone. After the silver "cake" is filtered and dried it can be dissolved into nitric acid to make a "technical" grade silver nitrate solution or it can be melted to give pellets or bars of silver.

Economics of this Method

By this method 59 g of silver-bearing electric contact scrap salvaged from military electronics scrap was dissolved with 125 mL of concentrated nitric acid and the precipitated silver chloride was dissolved into 200 mL of concentrated ammonia then precipitated with 15 g of ascorbic acid[4] to produce 28 g of silver after melting. The cost of the chemicals at the Fisher retail listing was estimated as \$2.10 to produce silver worth approximately \$5.40. This silver is also equivalent to 44 g of silver nitrate worth approximately \$45. The purity of the silver was estimated by dissolving 580 mg into minimum nitric acid followed by basification with concentrated ammonia. The resulting solution had a faint blue color of the copper ammine complex ion indicating a trace of copper present, probably that equal to impurity in 99.9% silver. More washing of the precipitates would likely have removed much of this contaminating copper.

Interferences

We have tested the efficacy of this method finding, in general, a 95–100% return of the theoretical amount when pure silver nitrate is the silver source. When other metal contaminants are present there would be the possibility of them interfering with the reduction or being co-precipitated. We have tested this method using significant amounts of nitrates of nickel, cobalt, cadmium, copper, zinc, and lead with the silver nitrate. We found no significant reduction in the yields of silver and no apparent changes in the precipitation. While the purity of the silver residues was not checked, we found that these silver residues melted with the appearance of silver free from any significant contaminants.

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[2] *Talanta* **1979**, *26*, 603.
[3] Multiple washings will improve the purity of the silver by washing away possible impurities.
[4] Available from Sigma Chemicals, St. Louis (phone: 800-325-3010) at approximately \$24 per kilogram.