41. Silver Nitrate from Silver Residues

This method of silver recovery can be applied to any silver precipitates from quantitative analysis, such as silver chloride, bromide, or thiocyanate. First wash the residues well with water by decantation and on the Büchner funnel, then spread out on paper to dry. A little nitrobenzene absorbed on the precipitate from the Volhard titration will not do any harm; the greater part of the nitrobenzene will be removed by washing, in any case.

Take 50 grams of dry residues, 100 grams of anhydrous sodium carbonate, 75 grams of potassium carbonate, and 25 grams of sodium or potassium nitrate. Grind them up together and mix well. Pack the mixture into a large Hessian fire-clay crucible, tamp down with a pestle, and fill the crucible right up to the top, putting in more mixture if necessary. Heat the crucible in a Fletcher gas furnace for 3 or 4 hours. A little caution is needed here, because the alkali carbonates in the crucible will attack the silicates of the crucible walls and may make a hole in the side of the crucible if the heat is too intense. The crucible should therefore be placed on the side of the furnace away from the hole where the flame comes in, the maximum heat possible with a full gas supply should not be used, and the bottom of the furnace should be covered over with sand beforehand to lessen the damage

to the furnace if a leak should occur. Ordinarily, however, the crucible will not fail.

At the end of the fusion, the mass in the crucible should be quiet, with no bubbling. When the crucible is cool enough to handle, break it open with a hammer and take out the silver button. Hammer away as much as possible of the adherent slag and flatten the button out somewhat, and weigh it.

Wash the button with a little 6M hydrochloric acid and water, then put it into a beaker with 75 ml of concentrated nitric acid and 50 ml of water; heat it on the steam bath, or, better, on a hot plate in the hood until all the silver has dissolved. Filter the solution if necessary. Evaporate on the steam bath or hot plate to about 75 ml; cool slowly, finally cooling in ice. Filter the crystals of silver nitrate on a Büchner funnel, preferably one with a sintered glass plate, suck as dry as possible, and spread the crystals out on a watch glass to dry. The solubility of silver nitrate is high, even at 0° (122 grams salt to 100 grams water), so it may be worth while to evaporate down the mother liquor further and obtain a second crop of crystals, which, however, will not be as pure as the first. The first crop, if dried well enough to remove adhering nitric acid, will normally be pure enough for most purposes. It can, of course, be recrystallized if desired. The purity of the salt should be checked by analysis, especially if the salt is to be used in the analytical laboratory.

Silver nitrate stains the fingers. Clean hands the day after this experiment are a sign of excellent technique.