CHAPTER XIII.—THE ANALYSIS OF THE METALLIC IMPLEMENTS AND PRODUCTS OF CORROSION.

In making choice of methods for the analysis of the metallic implements and products of corrosion, the purpose has been to secure results as accurate and comprehensive as may be with the least expenditure of valuable and unique material. In many cases the quantity of material employed did not exceed a half gram, and in some cases was less than that amount. The sample was taken from the implement in some cases by means of a lathe center-bit without disturbing exterior outlines, in some cases was broken from an end or edge, and in many cases was scraped or flaked off from the surface of the implement or broken from a mass of corroded material.

THE ANALYTICAL PROCEDURE.

I. Treatment of Material.

Of the material an amount approximating 0.5 gram, if so much was available, was carefully weighed, placed in a small covered beaker, and treated with 10 c.cm. of nitric acid (sp. gr. 1.42), the action being moderated by cooling when the sample was metallic and quickly acted upon, and accelerated by warming when the sample represented corroded material and was slowly attacked. When the action was complete, it was usual to evaporate the liquid, desiccate the residue at 110° C., moisten with nitric acid, take up soluble material in 50 c.cm. of water, treat with ammonium hydroxide to formation of the soluble copper-ammonium compounds, heat to boiling, add nitric acid in excess, and filter the insoluble material upon ashless paper and wash, reserving residue and filtrate for separate treatment.

By desiccating silica was made insoluble, and by treatment with ammonium hydroxide complete hydrolysis of soluble tin compounds was effected. In some of the earlier analyses of pure metal the process of desiccation was omitted, and for the formation of the insoluble metastannic acid reliance was placed upon dilution and boiling. Experience showed, however, that all the tin is found in the residue when the ammonium hydroxide treatment is employed, and this treatment was followed in nearly all the work.

II. The Insoluble Residue after Treatment with Nitric Acid.

The insoluble residue, consisting possibly of antimony oxide, metastannic acid, silica, undecomposed material, and lead sulphate in case lead and sulphur were present, was collected upon ashless paper, washed, ignited in porcelain, and weighed.

The residue was covered with a mixture of sodium carbonate and sulphur in equal parts, and the mass was heated in the covered crucible until the preliminary fusion was over and sulphur ceased to be volatilized. The mass was boiled