

of an analytical method is that with stannous chloride, which has been used by Plimpton<sup>1</sup> and by Russig<sup>2</sup> for the preparation of  $\alpha$ -hydro-naphthoquinone and its derivatives.

In the first experiments I treated the quinone with a large quantity of the stannous salt, and attempted to determine the excess of the latter with decinormal iodine. No satisfactory results were obtained, however, either at room temperature, or at  $100^\circ$ . This might be accounted for by the action of iodine on the hydro-naphthoquinone formed by the reduction; but even when the latter was removed the results were still unsatisfactory.

The plan was then changed, and the stannous salt was added to the quinone from a burette until the reduction was complete, the end point being recognized by the absence of characteristic color reactions of the quinone. Two methods of working were studied, differing in the nature of the indicator chosen; both gave good results, and in each  $2\text{SnCl}_4$  were required for one mol. of  $\text{C}_{10}\text{H}_6\text{O}_2$ .

*First Method:* A decinormal solution of stannous chloride in two per cent. hydrochloric acid<sup>3</sup> is run into the alcoholic solution of the quinone until the yellow color of the latter is almost discharged. The addition of the stannous chloride is then proceeded with drop by drop, testing after each addition on a watch glass, with a drop or two of a mixture of equal volumes of phenyl-hydrazine and alcohol. So long as a trace of the quinone remains a bright pink color is formed.<sup>4</sup> (Taken, 0.0700 g. quinone; found, 0.070 g.).

*Second Method:* To the alcoholic solution of the quinone, three or four drops of freshly distilled aniline are added, and the whole is boiled; whereupon it turns bright red.<sup>5</sup> Decinormal stannous chloride is then run in from a burette, heating to boiling after each addition, until the red color is completely discharged. If the end point has been over-stepped, a volumetric solution of the naphthoquinone may be used for titrating back. (Taken, 0.0700 g. quinone; found, 0.072 g.).

*Purification of  $\beta$ -Naphthoquinone:* This compound was prepared according to the method of Lagodzinski and Hardine<sup>6</sup>; about 40 g. of crude quinone being obtained from 50 g.  $\beta$ -naphthol. No method could be found in the literature for the purification of this crude material, and attempts to recrystallize it from ether, alcohol, benzene, petroleum ether, acetone, chloroform, or mixtures of these, were unsuccessful.

<sup>1</sup> J. Chem. Soc. **37**, 635 (1880).

<sup>2</sup> J. pr. Chem. **170**, 32 (1900).

<sup>3</sup> If the acid is too concentrated, some of Knapp and Schultz's amorphous compound may be formed. Ann. **210**, 175 (1881).

<sup>4</sup> Ber. **17**, 3026, (1884).

J. Chem. Soc. **37**, 635 (1880).

<sup>5</sup> Ber. **27**, 3075 (1894).