generated electrolytically from a solution of caustic potash. On standing, however, the solution turned brown, and gave results different from those with a fresh preparation. In the end, I found it best to make up a fresh solution daily; the concentration was determined by titration with normal sulphuric acid, using methyl orange as indicator.

Method of Working

The reagents were measured into large test-tubes (150 cc) closed with rubber stoppers, and placed in a thermostat at zero. A separate mixture was used for each analysis, for fear of introducing water. The sodium ethylate pipette was washed with water and dried with a hot air-blast before each measurement.

When it was desired to stop the reaction, a measured volume of water was added, whereupon the caustic soda (formed from the ethylate and from the sodium-acetonoxalic ester) promptly saponified the excess of oxalic ester, and was itself neutralized in the process; as one mol of the ester will thus neutralize two of the ethylate, it was not necessary to add additional ester except when an excess of ethylate had been used in making up the reacting mixture. After the caustic soda has been neutralized the ethyl oxalate is very slowly saponified, and thus makes the solution only slightly acid.

A known fraction of the aqueous alcoholic solution was then removed, mixed with 4 cc of the ferric chloride,¹ and compared with the standard in the colorimeter; enough of the reaction product was taken in each case to bring the reading between 30 and 50 mm. Two or three settings of the colorimeter were made in each case; and from three to eight samples of each reacting mixture were pipetted out and analyzed, depending on the agreement between the results, and on the ease with which I found I could decide on the end-point—that is to say, no doubt, on the state of my eyes.

¹ See foot-note, p. 7.