A VOLUMETRIC METHOD FOR THE DETERMINATION OF PHENOL-*p*-SULFONIC ACID.

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Formation of the dibromophenolsulfonic acids, by addition of bromine to water solutions of alkali phenolsulfonates, was first reported by Sennhofer.[1] We have found no record of the reaction involved having been used for analytical purposes. Our experiments with sodium and zinc phenol-*p*-sulfonates of known purity show that it is practically quantitative under conditions specified below and adapted to the titrimetric determination of phenol-*p*-sulfonic acid in some of its salts. The procedure we have adopted is similar to that of Koppeschaar's method for the determination of phenol. As strict adherence to various particulars is required for accurate results, we give the method herewith in full detail.

A quantity of sample corresponding to 0.18 to 0.2 gram of $C_6H_4.OH.SO_3H$ is dissolved in 50 cc. of water in a 250 cc. glass stoppered flask having a long and narrow neck. To this are added 50 cc. of a water solution containing 2.7833 g. of $KBrO_3$ and about 40 grams of KBr per liter. (This solution is, in effect, a 0.1 *N* Br solution when an excess of acid is added; the large excess of bromide tends to minimize vaporization of the excess of bromine in the determination.) Then 5 cc. of hydrochloric acid, sp. gr. 1.18–1.19, are added, the flask quickly stoppered to prevent loss of bromine, and the mixture allowed to stand at 20° to 25° for not less than 10 nor more than 15 minutes. (Longer contact with bromine soon causes formation of tribromophenol, shown by a turbidity or flocculent precipitate. Formation of a precipitate or turbidity within 15 minutes indicates presence of phenol or other substances in the sample that affect the accuracy of the method.) A solution of about 1 gram of potassium iodide in 5 cc. of water is at once added, best, to prevent loss of bromine, by pouring first a little of it upon the juncture of the flared neck of the flask and the stopper, while the latter is still inserted, then raising the stopper just high enough to quickly introduce the remainder. After thorough shaking in the closed flask the solution is titrated with 0.1 *N* $Na_2S_2O_3$. No indicator is needed, but starch solution can be used. The number of cc. required is subtracted from 50 cc. Each cc. of the remainder corresponds to 0.004353 gram of $C_6H_4.OH.SO_3H$.

The limit of error is $\pm 0.5\%$. When the approximate quantity of phenolsulfonic acid in a sample is not known, a rough preliminary determination should be made, as the excess of bromine must be within the limits indicated, when accurate results are expected.

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[1] *Ann.*, 156, 103.