

CATALYSIS IN VOLUMETRIC ANALYSIS.

PART III. IODIMETRIC ESTIMATION OF PERSULPHATE WITH FERROUS SULPHATE AS CATALYST.

By G. GOPALA RAO, J. V. S. RAMANJANEYULU and V. MADHUSUDANA RAO,
Andhra University.

(Communicated by Dr. N. R. Dhar, D.Sc., F.R.I.C., F.N.I., I.E.S.)

(Received October 22, 1945; read April 17, 1946.)

In a previous publication Gopala Rao and co-workers have shown that persulphate can be estimated with ease and rapidity by making use of the reaction between persulphate and potassium iodide with cuprous iodide as catalyst. These investigators have also reported that silver, mercury, cerous, cobalt, nickel and manganous salts have no catalytic action on this reaction either in neutral or acid solution. We have now made experiments to ascertain if the catalytic action of ferrous salts can be employed for the purpose of volumetric estimation of persulphate.

EXPERIMENTAL.

20 ml. of persulphate (approx. M/50) are kept in a stoppered bottle together with 20 ml. of M/4 potassium iodide and then a small volume of N/50 ferrous ammonium sulphate is added. The liberated iodine is estimated from time to time by titration with standard sodium thiosulphate solution (approx. N/10). The results are recorded in the following table:—

TABLE I.

20 ml. of persulphate solution + 20 ml. of potassium iodide solution.
Amount of persulphate taken is equivalent to 10.15 ml. of hypo-solution.

AMOUNT OF PERSULPHATE REACTED IN MLS. OF HYPO-SOLUTION.				
Time in minutes.	Without catalyst.	With 0.2 ml. of FeSO ₄ solution.	With 0.4 ml. of FeSO ₄ solution.	With 1.0 ml. of FeSO ₄ solution.
5	2.50	4.00	6.20	8.75
10	3.70	5.45	8.00	9.95
15	4.80	6.60	9.00	10.10
20	5.70	7.40	9.55	10.10
30	6.90	8.55	10.05	10.10
40	7.80	9.30	10.10	10.15
60	8.95	9.90	10.15	10.15

From these results it will be seen that an increase in the amount of catalyst increases the speed of the reaction. The reaction is complete in fifteen minutes

with 1.0 ml. of N/50 ferrous ammonium sulphate acting as the catalyst. From another series of experiments, where the concentrations of the potassium iodide and the catalyst are doubled we found that the reaction is completed in less than ten minutes. The reaction thus catalysed will, therefore, be very suitable for the iodimetric determination of persulphate. We carried out numerous determinations of persulphate by this method and the values obtained are in agreement with those secured by Le Blanc's method. A few typical results are given below.

TABLE II.

X ml. of persulphate solution + 10 ml. of potassium iodide + 20 ml. of FeSO_4 solution (M/50).
Time 15 minutes.

AMOUNT OF PERSULPHATE FOUND IN GRAMS.	
Authors' iodimetric method FeSO_4 catalyst.	Standard method. Le Blanc's method.
0.1110	0.1100
0.0828	0.0822
0.0551	0.0552
0.0276	0.0280
0.0532	0.0532
0.0397	0.0397
0.0135	0.0137
0.0252	0.0252

The method for the estimation of persulphate now developed is perhaps the most convenient of all the known methods. Unlike the cuprous iodide catalyst, the ferrous sulphate catalyst is very rapidly prepared and the time of reaction is much shorter.

In conclusion, we desire to express our thanks to Prof. N. R. Dhar, D.Sc. (Lond.), Dr.es' Sciences (Paris), F.R.I.C., F.N.I., for his kind interest in this investigation.

REFERENCE.

- Gopala Rao, G., Ramanjaneyulu, J. V. S. and Madhusudana Rao, V. (1945). Catalysis in Volumetric Analysis. Part I. Iodimetric Estimation of Persulphate. *Proc. Nat. Inst. Sci. India*, **11**, 331-333.