

motor lorries can ply as far as Sahasradhara and the main locality is hardly three miles by pony road from it.

Geology Department,
Lucknow University,
Lucknow,
November 8, 1944.

H. L. CHHIBBER.

1. On the one inch sheet 53J/3 this place is spelt as Sansa Dhara but the correct spelling is Sana,radhara.

CATALYSIS IN VOLUMETRIC ANALYSIS

Estimation of Potassium Persulphate

If a reaction is to serve as the basis of a volumetric analytical process, it must be a very speedy one. While ordinarily very fast reactions only are selected for the purpose, some of the slowly occurring reactions have also been utilized on account of their convenience otherwise, the speed of reaction being increased by elevation of temperature. Only a few cases are on record where the speed of a reaction is increased for analytical purposes by the use of a suitable catalyst. Recently Gopala Rao and Ramacharlu¹ have employed sunlight or artificial light to accelerate a reaction so that it becomes suitable for purposes of quantitative analysis. It appeared to us that the phenomenon of catalysis can be utilized to a fuller advantage in volumetric analysis than has been the case hitherto. We have found that a suspension of cuprous iodide in water serves as an excellent catalyst for the reaction between potassium persulphate and potassium iodide and that the reaction so catalysed at room temperature is quite suitable for the iodimetric estimation of persulphate. The iodimetric method has the advantage that it is simple and accurate, giving directly a measure of the oxidizing power of persulphate. The methods now in use, with the exception of the iodimetric method of L. von Zombory,² are cumbersome. The alkalimetric method is based on the well-known reaction $2S_2O_8^{2-} + 2H_2O = 4HSO_4^- + O_2$ which takes place rapidly at 100° C. This method is not suitable for the estimation of ammonium persulphate, as nitric acid and nitrogen are also obtained due to secondary reactions. Moreover, the method is vitiated by the presence of bisulphate in the original sample. The method of Le Blanc and Eckardt is an indirect one, being based on the fact that persulphates oxidize ferrous sulphate, the speed of the reaction being considerable when the latter is present in excess.

The cuprous iodide catalyst used in our experiments was prepared by adding a slight excess of potassium iodide to a known quantity of pure copper sulphate (Merck, A.R. sample) in dilute solution, washing the precipitate obtained repeatedly by decantation with water until free from all traces of free iodine. The cuprous iodide thus prepared was suspended in water and the suspension made to a known volume and preserved in a wide-mouthed glass-stoppered bottle. This was found to be quite stable for several months, no trace of iodine or cupric salt appearing. The suspension used in our experiments contained approximately 0.015 gm. of cuprous iodide per millilitre. The

results recorded in Table I demonstrate the catalytic action of cuprous iodide.

TABLE I

15 ml. of potassium persulphate solution +
20 ml. of 0.125 Molar potassium iodide solution
Amount of persulphate taken = 0.1014 gm.

Time in minutes	Amount of persulphate reacted	
	Without catalyst	With 2 ml of cuprous iodide suspension
10	0.01624 gram	0.08672 gram
20	0.02581 "	0.08692 "
40	0.03643 "	0.08754 "
60	0.04591 "	0.08816 "
80	0.05267 "	0.08869 "
100	0.05663 "	0.08900 "

The catalytic action of cuprous iodide has been applied to the volumetric determination of persulphate in the following manner. 20 mls. of the persulphate solution are placed in a glass-stoppered bottle or Erlenmeyer flask, 20 mls. of potassium iodide solution (M/2) are added, followed by 5 mls. of the cuprous iodide suspension. The bottle or flask is kept stoppered for ten to fifteen minutes and the iodine liberated is titrated with a standard solution of sodium thiosulphate. The results are given in column 1 of Table II. These compare very favourably with those in column 2, the latter being obtained by the method of Zombory.² For the estimation of persulphate by our method, it is desirable to have the iodide at a concentration, 20 to 50 times that of the persulphate.

TABLE II
Amount of Persulphate Found

Authors' method (15 minutes)	Zambory method (30 minutes)
0.13340 gram	0.13340 gram
0.10100 "	0.10100 "
0.03684 "	0.03656 "
0.05286 "	0.05299 "
0.01823 "	0.01828 "

Thus it will be observed that our method requires a much shorter time than that of Zombory. We have found that silver, mercuric, cerous, cobalt, nickel and manganous salts do not catalyse the reaction between persulphate and iodide either in neutral or acid medium, while ferrous and ferric salts are good catalysts.

G. GOPALA RAO.
J. V. S. RAMANJANEYULU.
V. MADHUSUDHANA RAO.

Andhra University, and
Andhra Christian College,
Guntur,
November 17, 1944.

1. Gopala Rao, G., and Ramacharlu, P. T., *Curr. Sci.*, 1942, 11, 102; *Proc. Nat. Inst. Sci. (India)*, 1942, 8, 383; *Ibid.*, 1943, 9, 67. 4. 2. Zambory, L., *Von. Z. Anal. Chem.*, 1928, 73, 217.