

Short Communication

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COLORIMETRIC DETERMINATION OF PEROXYDISULPHATE

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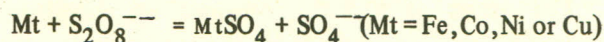
Alkali peroxydisulphates ($M_2S_2O_8$) can be determined accurately by volumetric methods involving redox reactions, viz. (a) Zn-EDTA method [1], (b) titration of $S_2O_8^{--}$ with standard hydrazine sulphate solution in the presence of redox indicator [2], (c) iodometric method [2-4], (d) reduction of $S_2O_8^{--}$ with an excess of standard Fe^{++} solution [5] followed by the determination of unreacted Fe^{++} reduction of $S_2O_8^{--}$ with an excess of standard Fe^{++} solution [5] followed by the determination of unreacted Fe^{++} (e) reduction of $S_2O_3^{--}$ as in (d) followed by the determination of Fe^{+++} by mercurous nitrate [5] and (f) reduction of $S_2O_8^{--}$ with an excess of standard oxalic acid solution [5] followed by the determination of unreacted oxalic acid.

The Zn-EDTA method [1] is the simplest of all the methods. This method involves stoichiometric reaction between Zn and $S_2O_8^{--}$ [1,6] followed by the determination of Zn^{++} by EDTA. Besides zinc, at room temperature, iron, cobalt, nickel and copper also dissolve in $S_2O_8^{--}$ solution according to the reaction represented by the

Table 1. Determination of $K_2S_2O_8$ by colorimetric and Zn-EDTA methods.

Concn of $K_2S_2O_8$ (mg/100 ml)	Concn of $K_2S_2O_8$ found (mg/100 ml)			
	Copper	Cobalt	Nickel	Zn-EDTA
0.1385	0.1390	0.1386	0.1383	0.1382
0.1385	0.1390	0.1388	0.1380	0.1382
0.1385	0.1380	0.1390	0.1387	0.1388
0.1385	0.1390	0.1388	0.1387	0.1382
0.2060	0.2070	0.2063	0.2057	0.2057
0.2060	0.2065	0.2063	0.2057	0.2060
0.2060	0.2055	0.2058	0.2066	0.2057
0.2060	0.2065	0.2066	0.2063	0.2063
0.3428	0.3420	0.3433	0.3436	0.3430
0.3428	0.3435	0.3426	0.3427	0.3425
0.3428	0.3425	0.3433	0.3427	0.3425
0.3428	0.3435	0.3423	0.3433	0.3425

following general equation.



The reaction between iron, cobalt, and nickel and $S_2O_8^{--}$ is appreciably fast while that between copper and $S_2O_8^{--}$ is slow. Nevertheless, the reaction between copper and $S_2O_8^{--}$ is accelerated in the presence of dil sulphuric acid containing a little Cl^- . These metals produce coloured ions which can be determined by colorimetric method, thus providing a means for the colorimetric determination of $S_2O_8^{--}$. However, iron cannot be employed for this purpose because the reaction between iron and $S_2O_8^{--}$ is uncertain; during the reaction primarily Fe^{++} is produced but subsequently it is partially oxidised to Fe^{+++} by excess $S_2O_8^{--}$ and atmospheric oxygen. In this communication colorimetric procedures for the determination of $S_2O_8^{--}$, using nickel, cobalt and copper are described.

EXPERIMENTAL

Procedure 1 - (Nickel and Cobalt Method). (a) Stock solution of nickel and cobalt was prepared by dissolving an accurately weighed quantity (0.1 - 0.2 g) of the metal powder in 20 ml 5M HCl and diluting the solution to 1000 ml in a volumetric flask. (b) To 50 ml aliquots of standard peroxydisulphate solution 1 g metal powder was added. The reactants were swirled for about 15 min and then filtered and washed directly into a volumetric flask. (c) Nitroso-R-salt method [5] was used to make calibration curve and to determine cobalt in the test solution (b). In the case of nickel, dimethylglyoxime colorimetric method [5,7] was used.

Cobalt can also be determined by thiocyanate method [10].

Procedure 2 - (Copper Method). (a) Standard copper sulphate solution was prepared by dissolving an accurately weighed amount of copper powder in concd. nitric acid followed by the addition of 50% sulphuric acid, then evaporating the solution to dense white fumes of SO_3 and finally diluting it to the desired volume. (b) To 50 ml aliquots of standard peroxydisulphate solution 5 ml 10M sulphuric acid and 0.5 ml 5M HCl were added followed by 1 g copper powder. The reactants were swirled for about 15 min and filtered and washed directly into a volumetric flask. (c) Copper tetramine colorimetric method [7 - 9] was employed to make calibration curve and to determine copper in the test solution (b).

Alternatively copper may be determined iodometrically [5].

The results of these procedures, given in Table 1, were compared with the Zn-EDTA method [1].

RESULTS AND DISCUSSION

The values of $K_2S_2O_8$, obtained by colorimetric procedures, are comparable with the values obtained by Zn-EDTA method. The experimental error in assayed values of $K_2S_2O_8$ is within permissible limit of $\pm 0.5\%$. If the solution of $K_2S_2O_8$ is dilute, i.e. weaker than 0.1N, longer periods are required for completion of the reaction between the persulphate and the metals. The H^+ (except in the case of copper) and OH^- , organic compounds and the reagents which reduce or decompose neutral peroxydisulphate solution interfere with these procedures.

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