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A new field method for the determination of dissolved oxygen in water.

by

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SUMMARY

A simple and convenient method for the determination of dissolved oxygen in fresh and salt water has been developed. The iron (III) formed from ferrous ethylenediamine sulfate (FES) is titrated with EDTA solution in the presence of salicylic acid indicator. Advantages over the Winkler method are: a) restandardisations of the titrant are unnecessary; b) the reagents used are neither corrosive nor hygroscopic and they may be applied in solid form; c) interferences by reducing substances are less.

INTRODUCTION

Of the titrimetric methods available for the determination of dissolved oxygen in natural waters, the method of Winkler [1]) is most widely applied. For use on board ship or in the field it has, however, several disadvantages such as the corrosiveness of the reagents, the elaborate precautions needed for work with polluted waters, and the instability of the thiosulfate titrant.

Recently [2]), a method has been proposed which avoids these disadvantages. The chemical reactions taking place during the determination can be summarised as follows: dissolved oxygen reacts with iron (II) to from iron (III) which is then titrated with EDTA solution in presence of salicylic acid as indicator. The oxydation-step takes place at a pH of 7.5, the titration is carried out at a pH of 2.5.

Reagents:

a) Ferrous ethylenediamine sulfate (FES): $Fe(H_2NCH_2CH_2NH_2)(SO_4)_2.2H_2O$. This must not contain iron III (to check this, add to boiling water some sodium salicylate, maleic acid and FES. No violet color should develop within a minute. If necessary, purify the preparation as follows: Prepare a concentrated solution of the impure salt in water acidified with one drop sulfuric acid, filter if necessary, add some salicylate and sufficient EDTA solution just to remove the violet color. Immediately precipitate with an equal volume of ethanol, filter by suction, wash with ethanol and dry in a stream of cold dry air.

b) Tris(hydroxymethyl)aminomethane (THAM); $(CH_2OH)_3CNH_2$.

c) Maleic acid; HOOC - CH = CH - COOH (cisform).

d) Sodium salicylate; $C_6H_4(OH)$ COONa.

e) Ethylenedinitrilotetraacetic acid disodiumsalt, (EDTA);

$$\begin{matrix} NaOOC & & COOH \\ & NCH_2CH_2N & \\ HOOC & & COONa \end{matrix} .2H_2O.$$ Solution 0.1 molar.

For ordinary purposes weigh 37.21 grams of the analytical grade salt, dried at 80°C, and make up to 1 litre with destilled water. For more precise standardisation procedures see Schwarzenbach[3]). Kept in a good closed, thickwalled polyethylene bottle the solution will keep indefinitely. 1 ml is equivalent to 0.8 mg O_2.

PROCEDURE

To a sample of water in a weighed wide-necked glass-stoppered bottle of volume about 100 ml add in the following order, 250 - 300 mg of sodium salicylate, 150 - 200 mg of FES and 80 - 120 mg of THAM. Immediately close the bottle, without enclosing air bubbles and mix by thorough shaking. (The intensity of the brown color of the 1 : 2 ferric salicylate chelate gives an idea of the amount of dissolved oxygen in the water). Open the bottle and immediately add 600- 900 mg maleic acid. The color changes into a deep violet (the 1 : 1 ferric salicylate chelate). Titrate in the neck of the bottle with EDTA solution until the last trace of violet has disappeared from the yellow-green color of the ferric-EDTA chelate.

DISCUSSION

The amounts of iron(III)compounds formed during the determination are equivalent to the amount of oxygen in the water. All these complexes are colored, hence a variety of ways to determine oxygen colorimetrically opens up there.

A convenient tool to carry out titrations in the field is a micrometer buret (R.G.I. Inc., 1 Great Neck Road, Great Neck, New York, imported in Europe by Touzard and Matignon, 3 Rue Amyot, Paris 5e. It consists of a syringe with a Teflon plunger, operated by a micrometer screw. Available are burettes of 2 ml divided into 0.002 ml. If used with 0.1 molar EDTA and 100 ml oxygen bottles they cover the range of 0-16 mg O_2/litre. The contents of the bottle can be stirred during the titration by rolling a spatula with a round shaft and an narrow blade between thumb and fore-finger.

The method is less subject to interference by reducing substances than the Winkler method, because the reaction mixture itself is of a reducing nature (Iron(III) ions are all firmly bound by an excess of

The precision of the method is slightly less than that of the Winkler method. Under comparable conditions standard deviations of 0.062 and 0.045 mg O_2/litre respectively were obtained for both methods.

Literature cited.

1) L.W. Winkler, Ber. 21 (1888) 2843.

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3) G. Schwarzenbach, Die komplexometrische Titration.
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