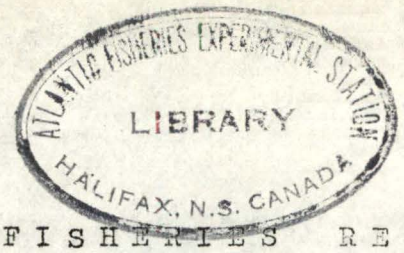

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Title

A FEW NOTES ON THE ANALYSIS OF PHOSPHATES IN SEA WATER BY THE DENIGES METHOD.

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A Few Notes on the Analysis of Phosphates in Sea Water by the Deniges Method.

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The reagents used in the Deniges method were prepared in the usual way. Reagent 1:- 11.3 grams ammonium molybdate were dissolved in 100 mls. distilled water at 60°C. One hundred and forty mls. C.P. concentrated sulphuric acid were diluted to 400 mls. and when both solutions had reached room temperature the ammonium molybdate solution was added to the sulphuric acid with constant stirring. When cool it was diluted to exactly 1 litre. Reagent 2:- 3.3 grams mossy tin were dissolved in 30 mls. concentrated C.P. hydrochloric acid which had been covered with a layer of benzene or toluene one quarter of an inch deep to exclude the air which would oxidize the stannous chloride. To facilitate the solution of the metal the reaction mixture was allowed to stand on a warm radiator over night. Two hundred and fifty mls. distilled water were boiled to expel any dissolved oxygen and allowed to cool under a layer of toluene. When cool it was added to the stannous chloride solution.

The method of analysis consists of adding 1 ml. of reagent 1, and 1 drop of reagent 2, to each 100-ml. sample of water. The mixture is thoroughly shaken and the color compared with a standard of known concentration after five minutes and before ten minutes standing. The standard is prepared from a stock solution containing 1 mg. PO_4 per litre by using from five to fifteen mls. of the stock diluted to 100 mls. as desired for the various concentrations encountered in water.

Several difficulties had been encountered when this method was applied to the analysis of sea-water samples on board ship. The first of these was that the standard did not develop the same shade of color as the sample, making it very difficult to compare. The usual standard solution was made up

in distilled water and when this procedure was varied by using artificial sea-water, a much better colour was obtained. This led to the use of either carefully analyzed sea-water or phosphate-free sea-water as a solvent for the standard phosphate. This method gave a very satisfactory color for comparisons.

It seemed advisable, also, to consider the optimum amounts of reagents that should be used for the various concentrations of phosphate that were encountered. This was done by using a representative low and a representative high concentration and adding increasing amounts of reagent 1 to the two series of tubes. After the requisite time had been allowed for the reaction, the colors were compared in a modified colorimeter against the strongest color developed. The results are given in Table I, and show that for any of the concentrations encountered, 1 to 2 mls. of reagent was the optimum quantity of the reagent. They also show that if too much reagent 1 is added, a yellow color develops which makes it impossible to match.

A similar series was carried out for reagent 2 and the results are given in Table II, showing that for all concentrations the optimum amount of stannous chloride is five drops instead of one, as recommended in the usual procedure.

Previous workers had stated that it was necessary to have the concentration of the standard very close to that of the sample. An investigation was undertaken to determine how close this approximation had to be made. This was done by making up a series of tubes containing from 0.01 to 0.32 mgs. PO_4 per litre and treating each in the same way with the optimum quantities of reagents 1 and 2. After standing for five minutes, the color developed by each tube was compared with that developed in each tube of higher concentration. In this way a series of curves was obtained with each successive concentration used as a standard. Figure I clearly shows the results obtained, and indicates that

the concentration of the standard must be extremely close to that of the sample or a very large correction must be applied to the reading. The curves are obviously linear but their slope is very markedly different from the theoretical value of 1 shown in the figure, hence the very large correction if the concentration of the standard is appreciably different from the samples.

Table I.

Volume Reagent ml.	Concentration 0.05	0.15
0.5	0.025	0.08
1.0	0.05	0.15
2.0	0.046	0.15
5.0	yellow	yellow
10.0	"	"

Table II

Volume of $SaCl_2$ (drops)	Concentration 0.05	0.15
1	0.026	0.08
2	0.042	0.128
3	0.048	0.144
4	0.049	0.145
5	0.05	0.15
6	0.047	0.141
7	0.045	0.134
8	0.043	0.129
9	0.039	0.120