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# THE DETERMINATION OF SILICATE IN SEA WATER

By

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The normal range of silicate in sea water is from 0.0 to about 200  $\mu\text{g}$ . atoms Si per liter at 20° C. It has not been fully demonstrated whether the silicate determined by the method described below is in the colloidal or ionic form or both.

A colorimetric method for the determination of silicate in mineral water was modified and made practical by Dienert and Wandenbulcke. Atkins was the first to apply the method to oceanographic investigations.

The slightly modified method of Dienert and Wandenbulcke is the most accurate and convenient for the estimation of such concentrations of silicate. By this method silicate with the proper hydrogen-ion concentration is converted by molybdate to a yellow-colored compound which is probably the heteropoly acid,  $\text{H}_8\text{Si}(\text{Mo}_2\text{O}_7)_2$ . Ferric and phosphate ions in sufficient concentration are known to interfere with the determination of silicate, but the concentrations of these substances in sea water is such that their effect is negligible.

## *Chemical Substances Required:*

1. Concentrated Sulfuric Acid (36 Normal)
2. Ammonium Molybdate,  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$
3. Potassium Chromate,  $\text{K}_2\text{CrO}_4$
4. Borax,  $\text{Na}_2\text{B}_4\text{O}_7\cdot 10\text{H}_2\text{O}$
5. Picric Acid,  $\text{C}_6\text{H}_2(\text{NO}_2)_3(\text{OH})$
6. Distilled Water

The sulfuric acid and ammonium molybdate reagents should be silicate-free. The distilled water used in the preparation of these reagents should also be silicate-free. Since silicate is extracted from glass by the distilled water, silicate-free distilled water should be stored only in nonglass containers.

Potassium chromate should be purified by recrystallization from water solution.

Picric acid often contains water and other impurities. To purify it, 40 g. are dissolved in hot benzene, separated from the water, and fil-

# THE DETERMINATION OF NITRITES IN SEA WATER

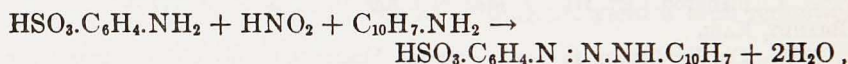
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The concentrations of nitrite-nitrogen in sea water varies from 0.000 to 2.00 microgram atoms ( $\mu\text{g. atoms}$ ) per liter of sea water at  $20^{\circ}\text{C}$ . In the open ocean nitrites are seldom found in depths below 100 meters, and even at these depths the water may be completely devoid of nitrites. When occurring in waters near the surface, the concentration is generally less than  $0.10\ \mu\text{g. atom}$  of nitrite-nitrogen per liter. The higher values are obtained at times in the waters on the continental shelf and in sounds, straits, fjords and estuaries.

The method recommended for the determination of nitrites, shown by the chemical equation



was originally developed by Peter Griess and later modified by Ilosvay. This method depends upon the diazotization of the amines in acid solution by the nitrite in the sea water. The product of this reaction is a rose-colored dye whose color intensity is proportional to the quantity of nitrite present.

### *Chemical Substances Required:*

1. Sulphanilic Acid,  $\text{HSO}_3\cdot\text{C}_6\text{H}_4\cdot\text{NH}_2$
2. Alpha-naphthylamine,  $\text{C}_{10}\text{H}_7\cdot\text{NH}_2$
3. Sodium Nitrite,  $\text{NaNO}_2$
4. Glacial Acetic Acid,  $\text{CH}_3\text{COOH}$
5. Mercuric Chloride,  $\text{HgCl}_2$
6. Distilled Water
7. Sodium Chloride,  $\text{NaCl}$
8. Chloroform,  $\text{CHCl}_3$

The above chemicals should all be pure substances and, with the exception of sodium nitrite, must be free of nitrites. The first five listed should be stored in bottles of amber glass with glass stoppers, while the other materials should be kept in glass-stoppered bottles made of resistant glass.

## REAGENTS

*Distilled Water.* Occasionally it is difficult to obtain distilled water free of nitrites. Should this be the case, suitable water may be secured by redistilling after making alkaline with sodium or barium hydroxide.

*Sulphanilic Acid.* One gram of the substance is dissolved by heating in a mixture of 15 ml. glacial acetic acid and 15 ml. of distilled water. More water is gradually introduced to the warm solution with constant stirring until 270 ml. of water have been introduced and the material has been dissolved. The solution should be stored in a glass-stoppered bottle of amber glass.

*Alpha-naphthylamine.* 0.2 g. of the substance is boiled with 40 ml. of distilled water. The colorless solution is decanted (while hot) from the dark-colored precipitate and treated with 15 ml. glacial acetic acid. This solution is diluted with 285 ml. distilled water. The solution should be stored in an amber, glass-stoppered bottle.

*Sodium Chloride.* 160 g. of sodium chloride are dissolved in 5 liters of distilled water and stored in suitable containers.

In place of sodium chloride solution, a nitrite-free sea water may be used. This water may often be obtained from the open ocean at depths below 200 meters. To prevent bacterial action, 5 ml. of a saturated solution of mercuric chloride are added per liter of sea water. This nitrite-free sea water is stored in the dark in demijohns or other suitable containers.

If nitrite-free sea water can not be obtained directly, it may be prepared by taking 4 liters of filtered sea water acidified with 0.5 ml. of glacial acetic acid and boiled for 45 minutes. The boiling operation is repeated on several successive days until the water is nitrite free. Evaporation losses are replaced by nitrite-free distilled water. To prevent bacterial action, 20 ml. of a saturated solution of mercuric chloride are added and the solution stored in a suitable container.

The rate of color development and not the final color intensity is affected by the presence of salts in solution. The development of the rose color with the reagents is much more rapid in sea water than in distilled water. Maximum intensity in the salt solutions and sodium chloride solutions is reached within 30 minutes, while with distilled water as much as two or three hours may be required. Increase in temperature likewise promotes the speed with which the reactions take place.

## PREPARATION OF STANDARD NITRITE SOLUTIONS

In the preparation of standard solutions, sodium nitrite is used. However, silver nitrite may be used, if a relevant technique is applied. The former compound is recommended in the preparation of the following solutions because of its greater convenience.

Emphasis must be given to the necessity for occasional check of all standard solutions.

The statements given below are based upon the experience of many investigators. However, unknown factors may influence the stability of the solutions, and possible faulty technique of the investigators may cause alterations in the concentration of the standard solutions. Therefore all standard solutions must be subjected to rigorous control which may best be accomplished by preparation of duplicate standards from time to time.

*Standard Solution I.* Sodium nitrite is recommended for the preparation of standard nitrite solutions. A few grams of pure sodium nitrite are dried for about four hours in an oven at  $110^{\circ}$  C. and cooled in a desiccator. Other adequate drying methods for the removal of hygroscopic water may also be used. 0.690 g. of the dried sodium nitrite is carefully weighed, transferred to a suitable container and dissolved in nitrite-free distilled water. The solution is transferred quantitatively to a liter volumetric flask, diluted to volume and thoroughly mixed. 0.2 ml. of chloroform or carbon disulfide is added as a preservative which insures a stable solution over many months. This solution is stored in tightly stoppered bottles and kept in the dark. Preparation of duplicate solutions is recommended. 1 ml. = 10  $\mu$ g. atoms nitrite-nitrogen.

*Standard Solution II.* 5 ml. of the Standard Solution I are pipetted into a 500 ml. volumetric flask, diluted to volume with nitrite-free distilled water and thoroughly mixed. 1 ml. of this solution contains 0.10  $\mu$ g. atom nitrite-nitrogen. This diluted standard solution is usually stable for several days. On longer standing the concentration may be lowered due to chemical or bacterial action. In some cases experience has shown that this solution may be stable for several months; however, good practice demands careful control.

*Standard Solutions III.* These standard solutions are prepared by diluting a certain amount (from 0.10 ml. to 1.00 ml.) of Standard Solution II to exactly 100 ml. with sodium chloride solution of nitrite-free sea water.

The number of standard solutions to be prepared depends upon the colorimetric method used. The standard solutions, given in Table I,

comprise those that are used when the colorimetric comparisons are made by means of Nessler tubes. If the colorimetric estimation is made with Hehner cylinders or with some form of colorimeter, then only one or two standard solutions are necessary.

The standard samples may be prepared and stored directly in the relevant containers for the colorimetric measurement (Nessler tubes, Hehner cylinders, etc.).

TABLE I. PREPARATION OF STANDARD SOLUTIONS III

Milliliters of Standard Solution II diluted to 100 ml.	Concentration of Standard Solutions III ( $\mu\text{g. atoms NO}_2\text{-N per}$ <i>liter of solution</i> )
0.00	0.00
.10	.10
.20	.20
.40	.40
.60	.60
.80	.80
1.00	1.00

Instead of sodium chloride solution or nitrite-free sea water, nitrite-free distilled water may be used for the preparation of the standard solutions for comparison with sea water from Standard Solution II, but sufficient time must be allowed for full color development.

### TESTING THE REAGENTS

When the standard solutions for comparison with the sea water are prepared, a blank determination should always be made to insure the absence of nitrites in the solutions used. This is accomplished by treating 100 ml. of the sodium chloride solution or nitrite-free sea water or distilled water with the reagents in the same manner as accorded to the standards and samples taken for analysis.

### THE PROCEDURE FOR THE DETERMINATION OF THE CONCENTRATION OF NITRITE-NITROGEN IN SEA WATER

The samples for the nitrite determination are treated with the reagents as soon as possible after they have been taken from the sea.

Whether the determination is made by means of Nessler tubes or by any other method, it is recommended that 100 ml. samples of sea water be taken for the determination.

Equal volumes of sulphanilic acid and alpha-naphthylamine acetate are thoroughly mixed. 4 ml. of the mixture are added to each standard

solution and sea water sample. The treatment of the standard solutions and the sea water samples must be the same. However, in cases where a number of samples are being collected over a period of days, the same standard solution for comparison may be used throughout a twenty-four hour period. Before the color comparisons are made, the temperatures of the standards and of the samples of sea water should not differ by more than  $2^{\circ}$  C. The comparisons of the samples with the standards may be made 30 minutes after temperature equilibrium has been obtained. If the color of one of the 100 ml. samples of sea water matches, for example, with that of the standard containing 0.60 ml. of Standard Solution II which has been diluted to 100 ml., then the sea water contains 0.60  $\mu$ g. atom nitrite-nitrogen per liter.

Should it be impossible to make color comparisons shortly after the sea water samples have been secured, the standards and the samples should be stored in the dark until conditions are more auspicious. The color is constant for four or five days. In any event, the mixture of reagents must be added as soon as the samples have been collected.

If difficulty is experienced in making color comparisons by means of Nessler tubes because of marked color intensity, then a smaller sample must be taken. A 50 ml. or even a 25 ml. sample of sea water is diluted with sodium chloride solution or nitrite-free sea water to 100 ml. in a Nessler tube and treated with 4 ml. of the mixed reagents. The nitrite-nitrogen concentration will be twice as great as indicated in Table I if a 50 ml. sample of sea water is used, and four times as great if a 25 ml. sample is taken.

*Accuracy of Results.* The accuracy of the determination is about 5 to 10 per cent. Because of the very small concentrations of the nitrites in sea water, no correction is necessary for the slight deviation in volume of sea water due to the deviation in temperature from  $20^{\circ}$  C. This statement also applies for the standard solutions used.

### APPARATUS REQUIRED

In accordance with the above description, the following solutions and apparatus are recommended for use on board ship. It is assumed that ample provision will be made for taking duplicate solutions and apparatus in case of accident or breakage. Duplicates of materials and solutions should always be packed in separate containers.

1. Apparatus for sampling the sea water.
2. Amber glass-stoppered bottles 100-250 ml. capacity with sulphanilic acid solutions.

3. Amber glass-stoppered bottles 100–250 ml. capacity with alpha-naphthylamine acetate solutions.
4. Amber glass-stoppered bottles 100–250 ml. capacity with Standard Solution I.
5. Bottles or demijohns of sufficient capacity for nitrite-free sea water, sodium chloride solution or distilled water.
6. Apparatus for preparation of standard solutions:
  - (a) 5 ml. pipet;
  - (b) 500 ml. glass-stoppered volumetric flask;
  - (c) Glass-stoppered bottles of 500 ml. capacity to contain the Standard Solution II;
  - (d) 5 ml. microburet, graduated to 0.05 ml., with buret stand;
  - (e) A suitable number of Erlenmeyer flasks, 125 ml. capacity, for preparation of the standard solutions for comparison with sea water according to the method used for the color comparison. If Nessler tubes are used for the color comparison, the standard solutions may be prepared directly in the Nessler tubes, thus making the Erlenmeyer flasks unnecessary.
7. Apparatus for mixing the solutions of sulphanilic acid and alpha-naphthylamine acetate solutions and applying the mixture to the standards and samples:
  - (a) 100 ml. graduated cylinder;
  - (b) 100 ml. bottle with a Mohr pipet, graduated to deliver 4 ml.
8. Apparatus for colorimetric measurements:
  - (a) 24 100 ml. Nessler tubes with graduation marks at the same height (diameter of 20 mm. may be convenient), or
  - (b) Hehner cylinders, or
  - (c) Colorimeter, or
  - (d) Photometer.
9. Thermometer graduated to 1° C.
10. Bottles with distilled water for cleaning the measuring apparatus necessary.

#### OTHER METHODS

The use of the dimethyl derivative of alpha-naphthylamine has been proposed, but it has no particular advantage. The diethyl derivative has been found to give a more stable color. The indol method, used many years ago, while giving fair results, is rather unsatisfactory because of the gradual decrease in color intensity after 45 minutes.



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tered to remove insoluble material. The benzene is evaporated under reduced pressure until water globules appear. The benzene solution is separated by decantation, and evaporation is continued until the picric acid is dry. The dry picric acid is stored in sealed vials until ready for use.

### REAGENTS

*Dilute Sulfuric Acid.* 5 ml. of concentrated sulfuric acid are diluted to 30 ml. with silicate-free distilled water.

*Ammonium Molybdate.* 10 g. of  $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$  are dissolved in 100 ml. of silicate-free distilled water.

*Borax Solution.* 10 g. of  $\text{Na}_2\text{B}_4\text{O}_7\cdot 10\text{H}_2\text{O}$  are dissolved to form a liter of solution.

### STANDARD SOLUTIONS

Solutions of sodium silicate, analyzed gravimetrically for their silicate content, have been used for the preparation of standards by dilution. This is a laborious process and the standards are stable for only limited periods. More permanent standards are easily prepared from either potassium chromate or picric acid solutions. Several investigators have determined the silicon equivalency of these solutions for this method of estimation. Experience has shown that a more readily reproducible standard solution may be prepared from a buffered solution of potassium chromate than from picric acid solution.

*Potassium Chromate Solution I.* 0.945 g. of  $\text{K}_2\text{CrO}_4$  which have been dried at  $105^\circ\text{C}$ . are dissolved in distilled water and diluted to 1 liter. 1 ml. of this solution is equivalent to 2.5  $\mu\text{g}$ . atoms Si.

*Potassium Chromate Solutions II.* These are prepared by diluting appropriate volumes of Standard Solution I to 50 ml. with 25 ml. of one per cent borax solution and distilled water. The concentration and number of standards II to be prepared are dependent upon the manner of comparison to be used and the concentration of silicate in the unknowns under investigation. In Table I are shown the dilutions when using Nessler tubes. For high concentrations of silicate, the 50 ml. tubes are recommended, while 100 ml. tubes are to be preferred for low concentrations.

*Picric Acid Standard Solution I (Alternate Standard).* 76.7 mg. of the pure, dried picric acid are dissolved in distilled water and diluted to a liter. 1 ml. of this solution is equivalent to 2.5  $\mu\text{g}$ . atoms Si, which is the same equivalency as the potassium chromate Standard Solution I; consequently it may be diluted as indicated in Table I.

TABLE I. PREPARATION OF STANDARD POTASSIUM CHROMATE SOLUTIONS II

Milliliters of Standard Solution I Diluted to:	Concentration of Standard Solutions II when Diluted to:	
	50 ml. ( $\mu\text{g. atom Si per liter of solution}$ )	100 ml.
0.20	10	5
.40	20	10
.60	30	15
.80	40	20
1.00	50	25
1.20	60	30
1.40	70	35
1.60	80	40
1.80	90	45
2.00	100	50
2.40	120	60
2.80	140	70
3.20	160	80
3.60	180	90
4.00	200	100

It seems to be generally agreed that in a sea water medium a given amount of silicate develops less yellow color with the reagents than it does in a distilled water medium. No such agreement exists concerning the magnitude of this "salt effect" or how it varies, if at all, with changing chlorinity. Consequently, the above standards have been left uncorrected for this factor.

### TESTING THE REAGENTS

The reagents should be tested occasionally for silicate impurities. The amount of reagents, as specified in the procedure, should be added to silicate-free distilled water. If at any time the reagents become contaminated with large amounts of silicate, new reagents should be prepared. If only small amounts are present, the appropriate correction can be deducted from each observed value when comparing the unknowns with the "permanent" standards.

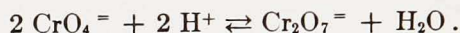
### THE PROCEDURE FOR THE DETERMINATION

The samples of water should be taken from the sampling bottles immediately after bringing them aboard ship. They should be taken directly in the Nessler tubes for analysis. When comparisons are to be made in a colorimeter or photometer, samples are drawn from the water bottle into a graduated cylinder, transferred to a 125 ml. Erlenmeyer flask. Plankton or sediment need not be removed from the sample when present only in small amounts. 0.5 ml. of ammonium molybdate reagent and 0.25 ml. of 6N sulfuric acid are added for each

50 ml. of sample. Comparison should be made with the standards not sooner than 5 to 10 minutes or longer than 1 hour after development of color.

### NOTES ON THE DETERMINATION

1. The borax solution acts as a buffer in the regulation of the pH of the solution. This is necessary to insure a constant equilibrium between the chromate and dichromate ions. The alkalinity of the buffer solution favors the formation of the yellow-colored ion.



2. The Chromate Standard Solutions II do not deteriorate on standing, but as a precaution new Solutions II should be prepared every six months. Solution I is stable indefinitely.

3. Picric Acid Standards are not as stable as the Chromate Standards. The ordinary C.P. picric acid contains varying quantities of water, and thus the compound should never be used without first removing the water and recrystallizing from benzene.

4. Picric Acid Standards should never be prepared by dilution with sea water as the dissolved salts have a decided "salt effect" upon the tautomeric equilibrium existing in the picric acid solutions.

5. If the samples are not analyzed immediately, they should be treated with the required amount of sulfuric acid in the Nessler tubes and then with the ammonium molybdate at a later time when the comparisons are to be made. In this manner samples may be preserved for about a day. It should be emphasized that comparisons with the standards should be made as soon as possible.

6. Under no circumstances should samples be stored in glass bottles for future analysis. Sea water acts rapidly on glass; therefore, samples that have been stored in glass containers for any length of time give high results and are valueless.

7. Samples containing large quantities of plankton or of sediment when stored in nonglass containers for any period of time give high results which are valueless.

8. Both the meta- and orthosilicates are analogous in their behavior toward the molybdate reagent, as might be expected, since in water solution orthosilicate has been shown to be converted to metasilicate.

9. A maximum error of 5 per cent occurs when Nessler tubes are employed, and this may be reduced somewhat by the employment of a colorimeter or photometer.

10. Nessler tubes which have been used for the phosphate determination should not be used for the determination of silicate, as color difficulties are often experienced if this is done.

11. A greenish tinge is sometimes obtained with water having more than 70  $\mu\text{g.}$  atoms Si per liter, especially when Nessler tubes are employed. This interference may be eliminated by the use of the photometer or by proper dilution of a smaller water sample.

12. Should it be deemed desirable to dilute samples high in silicates with distilled water, an analysis should first be made on the latter to ascertain its silicate content.

13. With waters of low silicon values, 100 ml. samples should be utilized. A sensitivity is obtained with Nessler tubes equivalent to 2 parts of Si in 100,000,000 parts of water.

14. Temperature has little effect upon the color intensity of the yellow heteropoly acid. However, the color development is slower at low temperatures, so that a longer time should be given. With a temperature of 5° C., half an hour is needed.

15. An excess of sulfuric acid causes a diminution in the color intensity, but no effect is noted with slight excesses of the molybdate reagent. The yellow color of the heteropoly acid reaches its maximum intensity within 5 minutes after the addition of the reagents and remains constant for nearly three hours. However, the samples should be compared with standards as soon as possible to avoid any increase in silicate concentration by the action of the sea water on the glass containers.

16. While the presence of compounds of iron and phosphorus may interfere with the determination, their occurrence in sea water is of such a concentration that any error from this source is negligible.

17. The silicon should be reported as microgram atoms of silicon per liter of water.

## APPARATUS AND SOLUTIONS REQUIRED

In accordance with the above description, the following solutions and apparatus are recommended for use aboard ship. It is assumed that ample provision will be made for taking duplicate solutions and apparatus in case of accident or breakage. Duplicate materials and solutions should always be packed in separate containers.

1. Apparatus for sampling sea water (sampling apparatus).
2. Amber glass-stoppered bottle, 100 ml. capacity, for 6N sulfuric acid.
3. Amber glass-stoppered bottle, 100 ml. capacity, for ammonium molybdate reagent.
4. Bottles of sufficient capacity for distilled water.
5. Metal containers of sufficient capacity for distilled water.

6. Glass-stoppered bottles of 500-ml. capacity for Standard Solution I.
7. 500 ml. glass-stoppered bottles for borax solution.
8. Apparatus for preparation of standard solutions and reagents:
  - (a) Balance accurate to 0.1 mg.;
  - (b) 100 ml. graduated cylinder;
  - (c) 25 ml. graduated cylinder;
  - (d) 1000 ml. volumetric flask, glass-stoppered;
  - (e) 5 ml. pipet, graduated to 0.1 ml.
9. Apparatus for measuring samples and reagents:
  - (a) If Nessler tubes are not used, an ample number of 125 ml. Erlenmeyer flasks made of Pyrex glass (or its equivalent) should be provided;
  - (b) 1 ml. pipet graduated to 0.01 ml.;
  - (c) 1 ml. pipet graduated to 0.1 ml.
10. 250 ml. beakers of Pyrex glass or equivalent, and 1000 ml. beakers of Pyrex glass or equivalent.
11. Apparatus for colorimetric measurement:
  - (a) Nessler tubes with graduation marks at same height (100 ml. tubes of 20 mm., and 50 ml. tubes of 16 mm. diameter, are convenient) or
  - (b) Hehner cylinders, or
  - (c) Colorimeter, or
  - (d) Photometer.

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