

A NOTE ON ANALYTICAL METHOD FOR SALINE LAKE WATER

Kunihiko WATANUKI,

*Department of Chemistry, College of General Education,
University of Tokyo, Komaba, Meguro-ku, Tokyo 153*

Tetsuya TORII,

Chiba Institute of Technology, Tsudanuma, Narashino 275

Shyu NAKAYA,

*Department of Geosciences, Faculty of Science, The University of Hirosaki,
Bunkyo-cho 3, Hirosaki 036*

Haruta MURAYAMA

*Faculty of Education, Yokohama National University,
156, Tokiwadai, Hodogaya-ku, Yokohama 240*

and

Sadao MURATA

Chiba Institute of Technology, Tsudanuma, Narashino 275

Abstract: For comparison of the analytical data a standard analytical method for saline lake water is proposed. In diluting the sample solution to make an adequate concentration for analysis, gravimetric sampling solution is recommended for the viscous high saline water. And to match the matrix of sample solution to the standard solution, the use of standard sea water is recommended.

1. Introduction

Chemical nature of lake waters in the Antarctic varies in total salinity and composition (WATANUKI, 1977). It is difficult to get a precise analytical result from the samples of such waters. It is desirable to make a standard analytical procedure for saline lake waters, in order to compare the data obtained by different researchers. The analytical method for saline lake waters is proposed in this paper.

2. Preparation of Sample Solution

In order to obtain a good result, a sample solution which has a suitable concentration for chemical analysis must be prepared. In ordinary water analysis, the

sample solution may be diluted with distilled water by using a pipette and a measuring flask. But in the case of highly saline water, the pipette is not adequate to take a definite volume of the solution, because the sample water is highly viscous.

The measurement of sample weight is necessary to obtain an accurate analytical result for a viscous solution. The procedure is as follows: Take an adequate amount of the sample solution in the flask with a stopper and weigh it. Then transfer it to the measuring flask and dilute it with distilled water. The resultant solution is stocked for chemical analysis. The analytical results must be presented in g/or mg per kg of the sample water, or at least the specific gravity of the original sample solution must be described so as to calculate the contents of chemical components in ppm.

First, the authors measure the specific gravity of the sample solution to know the dilution ratio, by using a specific gravity bottle, picnometer (25 or 50 ml) at 20°C or 25°C. Next, the authors prepare the sample solution by means of dilution as stated above, to adjust the specific gravity of the solution to that of sea water, because the authors use the standard sea water as an analytical reference in chemical analysis of elements. The specific gravity of sea water is 1.020~1.031, while the highest specific gravity found in Antarctica is 1.386 (Don Juan Pond in 1965), and an ordinary sodium-chloride type water has a value of 1.1~1.2.

3. Analytical Methods

Analytical methods that were employed in the field or in the laboratory are as follows.

Measurements of water pH and fixation of dissolved oxygen or hydrogen sulfide are carried out immediately after sampling.

Nutrient matters together with alkalinity and sulfide must be determined within several hours. Cations such as sodium, potassium, calcium, magnesium ions and anions such as chloride, bromide and sulfate ions are determined in the laboratory with the water samples stored in polyethylene bottles after brought to Japan.

Chalcophile elements, such as lead, zinc, cadmium and copper are determined with hydrochloric acid acidified water samples (pH=1).

3.1. Nutrient matters

Nutrient matters were determined by the methods which are principally based on the Manual of Oceanographic Observation (JAPAN METEOROLOGICAL AGENCY, 1970) and STRICKLAND'S Hand Book of Sea Water Analysis (STRICKLAND and PARSONS, 1968) with some modification.

Silicate-Si SiO_2 -Si: Molybdenum yellow method

Phosphate-P PO_4 -P: Molybdenum blue method

Ammonium-N NH_4 -N: Modified RICHARDS and KLETSCH method (1964)

Nitrate-N $\text{NO}_3\text{-N}$: Modified MORRIS and RILEY method (1963)

Nitrite-N $\text{NO}_2\text{-N}$: NISHIMURA's method (1969)

When hydrogen sulfide is present in the sample water, mercuric chloride solution is added to the sample to prevent the interference due to sulfide ions. After centrifuging the precipitate, the residual solution is analyzed as stated above.

3.2. Dissolved gas and alkalinity

Dissolved oxygen: Winkler Chemical Method is used. Electric DO-meter usually fails to give accurate results because of high salinity of lake water.

Hydrogen sulfide: Cadmium carbonate is added to the water in order to fix the hydrogen sulfide as cadmium sulfide and determine it by iodometry.

Alkalinity: Titrate with 0.1 N HCl to the pH of 4.3, using methyl orange or portable pH meter.

3.3. Cation

For a sodium-chloride type water, the standard sea water or an artificial sea water is recommended as a reference solution in order to match the matrix. For a calcium-chloride type water, dilution is effective.

Sodium: Low salinity-Atomic absorption spectrophotometry. High salinity-Gravimetry, measured as sodium uranyl zincacetate (BAKER and KOLTHOFF, 1928).

Potassium: Atomic absorption spectrophotometry.

Calcium and magnesium: Atomic absorption spectrophotometry or EDTA titration method.

If a large amount of magnesium is present, the titration with EDTA is difficult. In that case, after adding EDTA solution nearly to the end point, add the indicator and titrate to the end point. Also the use of EGTA (ethylene glycol bis (β -amino ethyl ether)-N,N'-tetraacetic acid) is recommended (SCHMID and REILEY, 1957).

3.4. Anion

Chloride: Low Salinity-Mercuric thiocyanate colorimetry method (IWASAKI *et al.*, 1956).

: High Salinity-Mercuric nitrate titration method (DOMASK *et al.*, 1952).

Sulfate: Low Salinity-Barium chromate colorimetry method (IWASAKI *et al.*, 1957).

: High Salinity-Gravimetry.

3.5. Others

Bromide: Iodometry (KAPLAN *et al.*, 1958).

Boron: Titration method using manitol (GAST *et al.*, 1958).

Chalcophile element: After extracted with MIBK and diethyldithiocarbamate, atomic absorption spectrophotometry is used.

This proposal is only tentative and trial, and the authors intend to develop

a new analytical method for saline lake water especially by using ion selective electrode.

References

- BAKER, H. H. and KOLTHOFF, I. M. (1928): A specific reagent for the rapid gravimetric determination of sodium. *J. Am. Chem. Soc.*, **50**, 1625–1631.
- DOMASK, W. G. and KOBE, K. A. (1952): Mercuric determination of chlorides and water soluble chlorohydrines. *Anal. Chem.*, **24**, 989–991.
- GAST, J. A. and THOMPSON, G. (1958): Determination of alkalinity and the borate concentration of sea water. *Anal. Chem.*, **30**, 1949–1951.
- IWASAKI, I., UTSUMI, S., HAGINO, K. and OZAWA, T. (1956): A new spectrophotometric method for the determination of small amounts of chloride using mercuric thiocyanate method. *Bull. Chem. Soc. Jpn.*, **29**, 860–864.
- IWASAKI, I., UTSUMI, S., HIGANO, K., TARUTANI, T. and OZAWA, T. (1957): Spectrophotometric method for the determination of small amounts of sulfate ions. *Bull. Chem. Soc. Jpn.*, **30**, 847–851.
- JAPAN METEOROLOGICAL AGENCY (1970): *The Manual of Oceanographic Observation*.
- KAPLAN, D. and SCHERS, I. (1958): Semimicro determination of bromides application to physiological fluids. *Anal. Chem.*, **30**, 1703–1705.
- MORRIS, A. W. and RILEY, J. (1963): Determination of nitrate in sea water. *Anal. Chim. Acta*, **29**, 272–279.
- NISHIMURA, N., MATSUNAGA, N. and KANAZAWA, H. (1969): Condition for coloration of nitrite with Griess-Romijin reagent and elimination of interferences of sulfur compounds and iodine. *Japan Analyst*, **18**, 1372–1376.
- RICHARDS, F. A. and KLETSCH, R. A. (1964): Recent researches in the field of hydrosphere, atmosphere and nuclear geochemistry. *Ken Sugawara Festival Volume*. Tokyo, Maruzen, 65–81.
- SANO, M., NAKAI, N. and TORII, T. (1977): Nurume Ike no biryo kinzoku no enchoku bunpu (Vertical distribution of some trace metals in Lake Nurume, Antarctica). *Nankyoku Shiryô (Antarct. Rec.)*, **58**, 108–115.
- SCHMID, R. W. and REILEY, C. N. (1957): New complexon for titration of calcium in the presence of magnesium. *Anal. Chem.*, **29**, 264–268.
- STRICKLAND, J. D. H. and PARSONS, T. R. (1968): *A Practical Handbook of Sea Water Analysis*. *Bull. Fish. Res. Bd. Canada*, **167**, 311 p.
- WATANUKI, K., TORII, T., MURAYAMA, H., HIRABAYASHI, J., SANO, M. and ABIKO, T. (1977): Geochemical features of Antarctic lakes. *Nankyoku Shiryô (Antarct. Rec.)*, **59**, 18–25.

(Received May 2, 1979)