NOAA Technical Memorandum NOS 30

COMPARISON OF RESULTS OF UNITED STATES
AND SOVIET DISSOLVED OXYGEN MEASUREMENTS
IN WATER BY THE WINKLER METHOD

Rockville, Md.
October 1984

U.S. DEPARTMENT OF COMMERCE / National Oceanic and Atmospheric Administration / National Ocean Service

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COMPARISON OF RESULTS OF UNITED STATES
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IN WATER BY THE WINKLER METHOD

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US/USSR Working Group of Experts on
Intercalibration and Standardization of
Oceanographic Instrumentation and Methods

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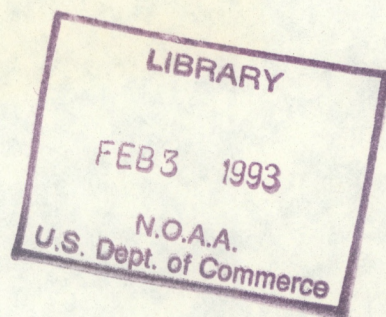
FOREWORD

Acknowledging the importance of the most complete possible study and effective utilization of the world ocean, the governments of the United States and the Union of Soviet Socialist Republics on June 19, 1973 concluded an agreement for cooperation in the area of research in the world ocean. This agreement, in particular, calls for intercalibration and standardization of oceanographic instruments and methods. After completion of intercalibration of three oceanographic parameters -- temperature, conductivity/salinity and current speed -- the joint working group considered it important to perform similar studies for the determination of oxygen dissolved in seawater.

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ABSTRACT. A cooperative program for determining the compara-
bility of oxygen measurements using a modified Winkler method
was conducted in Gelendzhik, USSR. The program involved
joint determinations of normality corrections of the standard
solutions. Joint measurements of dissolved oxygen content
under saturated conditions, in distilled water and seawater,
were conducted. Also, measurements were made with natural
(Black Sea) seawater at high and moderate dissolved oxygen
levels with emphasis at low and near zero concentrations.

INTRODUCTION

For approximately 100 years the dissolved oxygen content of water has
been determined by the volumetric iodimetric method suggested by L. W.
Winkler[1]. Since then the method has undergone many alterations, a number of
modifications have been suggested, but none have changed its basic nature.
The conditions under which analyses are performed have changed: the
concentration of reagents, volumes of samples, shapes of oxygen flasks, and
methods of determination of the titration end point.

In addition to the Winkler method, other methods are also used to
determine the quantity of oxygen dissolved in water: electrochemical,
gasometric, gas chromatographic, mass spectrometric, and other methods.
However, these measurements are still standardized and calibrated by means of
the Winkler method.

In terms of the number of observations in the world ocean, determinations of dissolved oxygen occupy third position after temperature and salinity, and frequently exceed the number of determinations of other chemical elements. When processing data on dissolved oxygen obtained by various expeditions, the researcher must consider the accuracy and reproducibility of each modification of the Winkler method, as well as the possible error in measurement results. For this purpose, methodological studies have been performed comparing the various modifications most commonly used in the US and England[2]. The sources of error in the Winkler method have been studied in detail, certain recommendations have been made and a number of technical improvements designed to increase the accuracy of analysis. These studies have been used by various researchers to develop new modifications of the Winkler method for hydrochemical research.

The present report includes the results of intercomparison of dissolved oxygen determinations by the Winkler method. The comparison measurements were conducted in October, 1981 in the USSR at Gelendzhik, using the facilities of the Southern Division of the P. P. Shirshov Institute of Oceanology, USSR Academy of Sciences, following a suggestion by the US-USSR Working Group of Experts on Intercalibration and Standardization of Oceanographic Instruments and Methods (Working Group 4).

The American side was represented by Dr. J. L. Solomon, Director of the Engineering Support Office, Office of Ocean Technology and Engineering Services, NOAA, and Co-Chairman of Working Group 4; and, also from the Engineering Support Office, A. N. Kalvaitis, Project Engineer, and J. P. Sullivan, Marine Chemist.

On the Soviet side, the following persons took part in the work: Dr. V. A. Shirey, Co-Chairman of the Soviet side of Working Group 4 and fellow at the Biohydrochemistry Laboratory, P. P. Shirshov Institute of Oceanology, USSR Academy of Sciences; Dr. A. M. Chernyakova, the group leader; P. A. Stunzhas; Yu. R. Nalbandov; and S. G. Poyarkov.

The intercomparison program developed at the Laboratory of Biohydrochemistry of the Institute of Oceanology was coordinated with the American side and had as its primary purposes the comparison of results of determination of oxygen dissolved in water using the standard methods of expeditionary hydrochemical research in the US and USSR. Primary attention

was given to the reliability of the results of dissolved oxygen determination at low and near-zero concentrations. The program included both laboratory and field experimental phases. The laboratory studies consisted of two sections: (a) determination of the correction factor for normality of the sodium thiosulfate solution (titer) and (b) determination of the content of dissolved oxygen in distilled water and seawater (at a given temperature and salinity) in equilibrium with the atmosphere (at a given pressure and 100 percent relative humidity). The field phase of the experiment included the collection of seawater samples of various dissolved oxygen values, treatment of the samples to preserve the oxygen level and delivery of the samples to the shore laboratory for analysis.

METHODS AND EQUIPMENT

The Soviet analytical chemists used a modification developed by A. M. Chernyakova[3], while the American side used a micromethod suggested by J. H. Carpenter[4]. The participants in the intercomparison used their standard equipment and reagents.

The characteristics of the equipment used and concentration of the reagents used are presented in Tables 1 and 2, respectively.

MEASUREMENT RESULTS

A. Correction Factor for Sodium Thiosulfate (Titer) Solution Normality

Each analyst determined the titer of the sodium thiosulfate solution which he prepared using 10 determinations, utlizing the standards of both sides. The results are presented in Table 3.

It follows from Table 3 that, in spite of the difference in procedures for preparation of standard KIO_3 solutions, as well as the differences in titration methods, the difference in thiosulfate titers was statistically insignificant: the ratio of the correcting coefficients for the titers was 1:1.000 ± 0.001.

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TABLE 1. : Equipment Used for Measurement of Dissolved Oxygen

Equipment	USSR	US
Oxygen flasks	Erlenmeyer flasks, 120-140 ml, elongated NS-29 stopper, calibrated to 0.01 ml	Erlenmeyer flasks, 130-145 ml, slightly elongated stopper, calibrated to 0.01 ml
Burettes	Volume to 10 ml, automatic zero setting, resolution of 0.02 ml, calibrated	Volume to 0.8 ml, piston microburette (Manostat Co.), 0.1 μl digital resolution
Standard pipettes	1 and 10 ml, calibrated to 0.001 ml	1 and 10 ml, calibrated to 0.02 ml
Pipettes for fixing reagents	Automatic acrylic syringe pipettes, volume to 2 ml, calibrated to 0.1 ml	Automatic glass pipettes, volume to 2 ml, calibrated to 0.1 ml

TABLE 2. : Reagents Used for Measurement of Dissolved Oxygen

Reagents	USSR	US
Manganese chloride	500 g/liter	600 g/liter
Alkaline iodide reagent	(700 g KOH + 300 g KI) per liter	(320 g NaOH + 600 g NaI) per liter
Sodium thiosulfate	0.02N	0.28N
Sulfuric acid	20 percent by volume	10N
Starch	0.5 percent	1 percent
Potassium iodate	0.02N (Salt dried 1 hour at 105°C)	0.01N (Salt dried 1 hour at 110°C)

TABLE 3.

Determination of Sodium Thiosulfate Titer Correction Coefficient

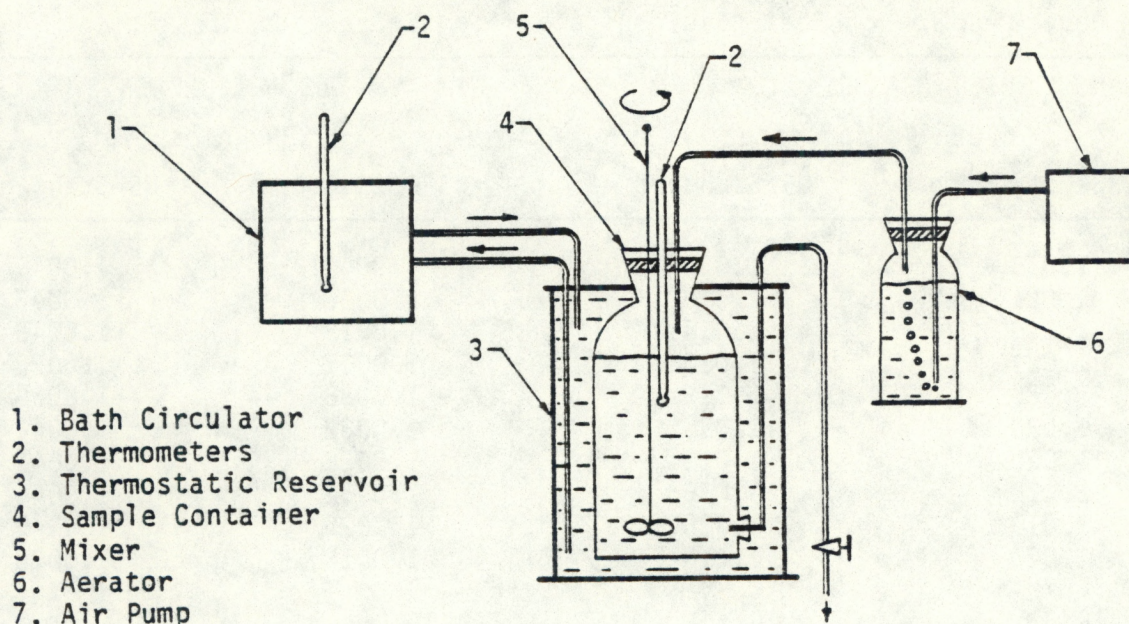| | USSR MEASUREMENTS 0.02N Thiosulfate | | US MEASUREMENTS 0.28N Thiosulfate | |
	USSR Standard	US Standard	USSR Standard	US Standard
1.	1.019	1.020	1.0041	1.0049
2.	1.021	1.018	1.0032	1.0043
3.	1.019	1.016	1.0018	1.0046
4.	1.021	1.020	1.0035	1.0052
5.	1.020	1.018	1.0038	1.0038
6.	1.020	1.020	1.0032	1.0041
7.	1.020	1.018	1.0038	1.0041
8.	1.019	1.016	1.0032	1.0046
9.	1.019	1.020	1.0029	1.0043
10.	1.020	1.018	1.0018	1.0038
$n =$	10	10	10	10
$\bar{x} =$	1.0198	1.0184	1.00313	1.00437
$\sigma =$	0.0008	0.0017	0.00079	0.00046

B. Determination of Oxygen Content in Distilled Water and Seawater in Equilibrium with the Atmosphere

A five liter bottle was filled with the water to be analyzed and immersed in a constant temperature water bath. A thermometer was placed in the bottle to measure the water temperature, as well as a mechanical stirrer and a hose to exchange the air above the water. An aquarium pump fed in the air through a bubbler (Figure 1) to adjust the humidity of the air to 100 percent. Before water samples were taken, the water in the bottle was agitated for three to four hours (agitation without formation of air bubbles). The water temperature in the bottle was maintained constant with an accuracy of better than ±0.1°C. The equilibrium oxygen content was computed from the International Tables of Oxygen Solubility[5] considering variations in atmospheric pressure during the experiment.

The parameters of the two samples were:

	Sample 1	Sample 2
Salinity :	0 parts/thousand	18 parts/thousand
Temperature :	24.0°C	22.0°C
Barometric Pressure :	763 mm Hg	762 mm Hg
O_2 Solubility :	5.90 ml/liter	5.51 ml/liter

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FIGURE 1. Apparatus for Atmospheric Equilibration

1. Bath Circulator
2. Thermometers
3. Thermostatic Reservoir
4. Sample Container
5. Mixer
6. Aerator
7. Air Pump

Samples were taken for analysis through the bottom tube of the bottle alternately by the American and Soviet analysts. The sampling procedure and the procedure for fixing dissolved oxygen were as outlined in the corresponding references[3,4]. The quantity of acid to dissolve the precipitate (about 1.3 ml) was selected so that the solution pH was approximately 2.1 for both analysts. The dissolved oxygen in the samples was calculated using the following equation:

$$O_2 \text{ (ml/liter)} = \frac{(V - V_b)(V_s)(N)(E)}{(V_2 - V_B)(V_3 - 2)} - O_x$$

where V is the reading from the burette considering the scale correction,

V_b is the blank correction,

V_s is the volume of the standard solution,

N is the normality of the standard solution,

E is the oxygen equivalent, 5598 ml O_2/eq,

V_2 is the mean volume of the thiosulfate solution used for titration of the standard,

V_3 is the volume of the oxygen flask,

2 is the volume of the sample replaced upon addition of the reagents,

and O_x is the oxygen content introduced with the reagents (0.018 ml/liter).

Since several of the variables were constant for the series of measurements (V_s = 10 ml, N = 0.01, E = 5598 ml O_2/eq, O_x = 0.018 ml/liter), the values can be inserted in the above equation in order to simplify:

$$O_2 \text{ (ml/liter)} = \frac{(V - V_b)(559.8)}{(V_2 - V_b)(V_3 - 2)} - 0.018$$

We can see from Table 4, which has the individual measurement results, that the best agreement was obtained for distilled water, where the standard deviation of results was not over 0.02 ml/liter and the results of the Soviet and American analysts fell within these limits, as did the theoretical concentration of oxygen in the sample. In the series of measurements by the Soviet analyst, two readings were discarded: No. 5, since its deviation from

TABLE 4.

Determination of Oxygen in Distilled Water and Seawater in Equilibrium the Atmosphere, with Dissolved Oxygen Expressed in ml/liter

	Distilled Water		Seawater	
	USSR	US	USSR	USA
1.	5.86**	5.89	5.43	5.50
2.	5.92	5.94	5.46	5.04*
3.	5.93	5.94	5.47	5.54
4.	5.91	5.91	5.44	5.42
5.	5.98**	5.89	5.46	5.30
6.	5.91	5.95	5.43	5.49
7.	5.90	5.90	5.45	5.51
8.	5.90	5.91	5.43	5.37*
9.	5.92	5.94	5.51**	5.26
n =	8	9	8	7
$\bar{x}$ =	5.91	5.92	5.45	5.43
σ =	0.02	0.02	0.02	0.11
Oxygen Solubility = 5.90			5.51	

 * Results rejected due to presence of air bubbles in oxygen flask
** Results rejected because deviation more than 2σ from the mean

the mean of the nine determinations exceeded two standard deviations (2σ); and No. 1, since its deviation from the mean of the remaining eight determinations exceeded 2σ. The difference in mean values between the analysts was tested for statistical significance according to the method of Appendix A; the test showed that the distilled water difference was not significant.

For seawater, the difference in mean oxygen concentrations was also 0.02 ml/liter, but σ for the American analyst was large -- 0.11 ml/liter. Judging from the results of determinations by the Soviet analyst, this sample of water had not reached equilibrium with the air, which may be related to the presence of small planktonic organisms. Since this sample of sea water was taken at a depth of 5 m, and filtered only through a realtively coarse screen, this could possibly account for the large σ in the US results.

C. Determination of Oxygen Content in Samples of Seawater

Seawater samples were taken in the aerobic zone of the Black Sea in an eight liter vinyl plastic water sampler. This portion of the experiment had two purposes: (a) determination of the oxygen content in samples of water with various oxygen concentrations and (b) determination of the content of oxygen in samples with low oxygen concentration. In the first case the water samples were taken at depths of 41, 91, and 130 m. On board the ship the same observer alternately took samples of water from each water sampler and fixed the dissolved oxygen in each sample by the methods used by the US and USSR. The samples were then delivered to the laboratory on shore for further analysis, performed by the American and Soviet analysts. Table 5 presents the results of these determinations. The test of Appendix A resulted in a statistically significant difference between analysts for only the 91 m sample.

For the second case, the samples were taken at depths of 110, 120, and 140 m, since low oxygen content with no free hydrogen sulfide was expected at these depths. The water from each water sampler completely filled a (capped) five liter bottle and the bottles were delivered to the laboratory onshore within one hour. The samples were taken by the analysts alternately; eight samples were withdrawn from each bottle. To avoid changes in the content of oxygen in the water due to contact with the atmosphere during sampling, a

TABLE 5.

Determination of Oxygen Content In Seawater Samples, in ml/liter

	41 m		91 m		130 m	
	USSR	US	USSR	US	USSR	US
1.	6.40	6.13**	5.05	5.01**	0.62	0.70
2.	6.40	6.36	5.09	5.12	0.64	0.54
3.	6.39	6.42	5.07	5.10	0.63	0.65
4.	6.41	6.38	5.07	5.10	0.68	0.59
5.	6.40	6.38	5.05	5.10	0.69	0.58
6.	6.39	6.42	5.04	5.10	0.69	0.59
7.	6.40	6.39	5.06	5.14	0.80	0.64
8.	6.40	6.42	5.07	5.08	1.26**	0.80**
n =	8	7	8	7	7	7
$\bar{x}$ =	6.40	6.40	5.06	5.11	0.68	0.61
σ =	0.01	0.03	0.02	0.02	0.06	0.05

** Results rejected because deviation more than 2σ from the mean

slight excess pressure was maintained above the water in the bottles by introduction of pure nitrogen. The results of the determinations are presented in Table 6. For this group of measurements, the test for differences showed the results for the 120 m sample to be significant.

In summary, the Soviet - American work showed good agreement of oxygen concentrations determined by the researchers of the two nations in the practice of expeditionary hydrochemical research. Considering all comparisons (Tables 4, 5, and 6), only two samples (91 m and 120 m levels) resulted in statistically significant differences between the analysts. The difference in the dissolved oxygen values in the other water samples may be attributed to the random errors inherent to the measurement process; these differences are within expected values for the modified Winkler methods used by both analysts for dissolved oxygen determinations. The differences are plotted in Figure 2; the vertical lines represent the bounds of statistical insignificance.

These comparison measurements have helped to select methodological concepts required for standardization of marine chemical measurements. It is considered important and timely to continue intercalibration of hydrochemical methods, primarily methods of determination of biogenous elements in seawater.
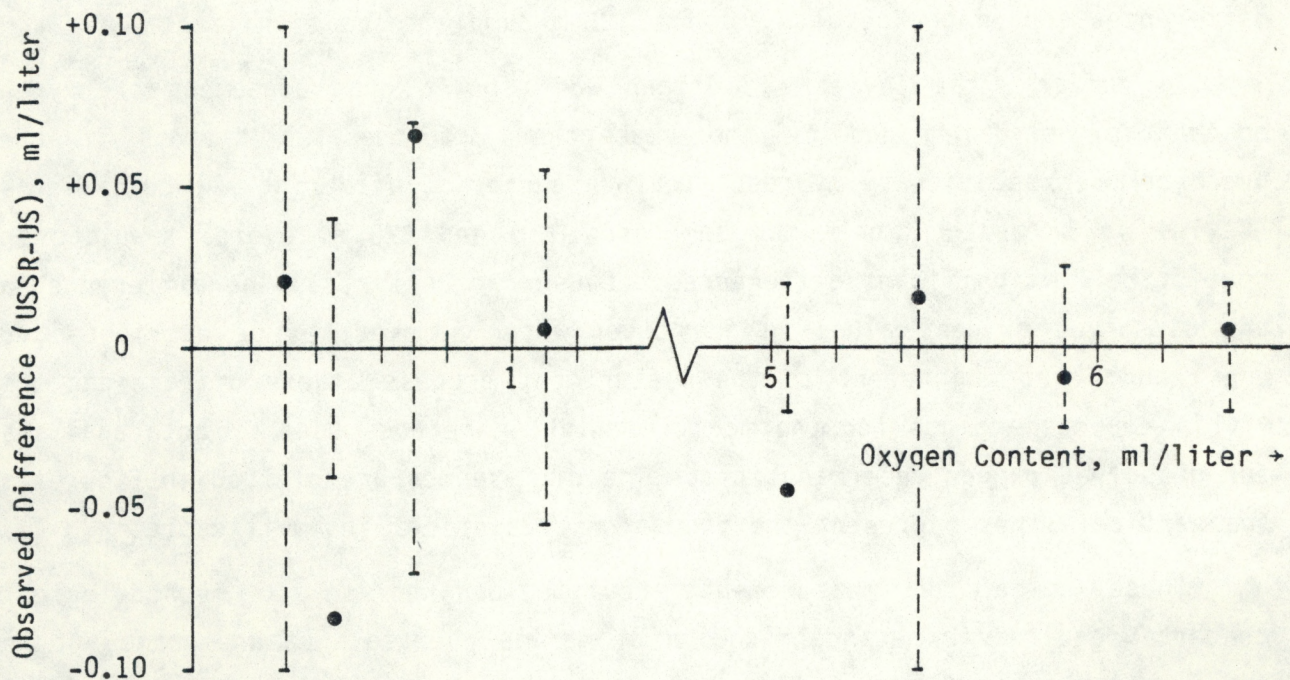
TABLE 6.

Analyses of Seawater Samples with Low Oxygen Content, in ml/liter

| | 110 m | | 120 m | | 140 m | |
	USSR	USA	USSR	USA	USSR	USA
1.	1.07	1.03	0.31*	0.50	0.53*	0.39
2.	1.11	1.10	0.36	0.46*	0.49*	0.38
3.	1.08	1.11	0.39	0.49	0.40*	0.25
4.	1.17	1.07	0.37	0.48	0.39*	0.26
5.	1.14	1.16	0.36	0.47	0.32	0.44
6.	1.09	1.21	0.41	0.47	0.36	0.28
7.	1.14	1.17	0.41	0.50*	0.39	0.23
8.	1.18	1.26*	0.47	0.50*	0.29	0.21*
n	8	7	7	5	4	7
$\bar{x}$	1.12	1.12	0.40	0.48	0.34	0.32
σ	0.04	0.06	0.04	0.01	0.04	0.08

* Results rejected because of air bubbles present in the oxygen flask

FIGURE 2.

Dissolved Oxygen Measurement Differences

ACKNOWLEDGEMENT

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APPENDIX A

Method for Testing Significance of Differences Between Analysts

In evaluating the differences in mean oxygen content resulting from the measurements of the US and Soviet analysts, it is important to decide whether the observed differences exceed what would normally be expected based on the expected imprecision of the analytical methods and equipment used. For this purpose, a statistical test was selected[6] to evaluate the observed differences between the two researchers.

In applying the test, it is first necessary to choose the significance level of the test (α) and to compute the combined degrees of freedom (ν) and combined sample standard deviation (σ_c):

Choose $\alpha = 0.05$

$\nu = n_1 + n_2 - 2$

$$\sigma_c = \left[\frac{(n_1 - 1)(\sigma_1{}^2) + (n_2 - 1)(\sigma_2{}^2)}{\nu} \right]^{0.5}$$

Next, the level of significant difference (u) is computed:

$$u = t\sigma_c[(n_1 + n_2)/(n_1 n_2)]^{0.5}$$

If the absolute difference between the observers, that is $|x_1 - x_2|$, is greater than u it is concluded that the observed difference is statistically significant and cannot be attributed to expected random differences.

In the above equations, the subscript "1" refers to the Soviet analyst and the subscript "2" refers to the US analyst. Symbols used are defined as follows:

    n is the number of samples used in computing the mean.

    x is the computed mean oxygen content.

    σ is the sample standard deviation of the mean.

    t is the value of the Student "t" distribution for ν degrees of freedom at the $(1 - \alpha/2)$ significance level.

(Continued from inside front cover)

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