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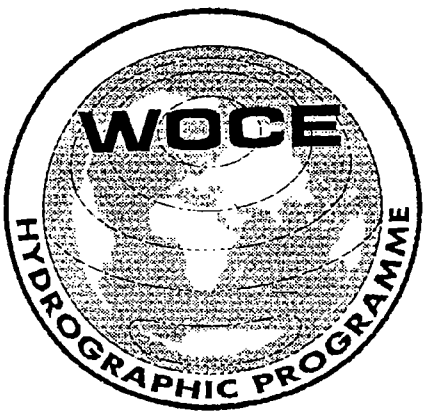

A Comparison of Methods for the Determination of Dissolved Oxygen in Seawater

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A Comparison of Methods for the Determination of Dissolved Oxygen in Seawater

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ABSTRACT

An intercalibration of dissolved oxygen methods was conducted at 2 stations in the Sargasso Sea between April 28 and May 3, 1990. The experiment compared three techniques using automated endpoint detection with the manual Winkler method using a starch endpoint. Institutions participating in the intercomparison were the Bedford Institute of Oceanography (automated photometric titration), the University of Delaware (automated amperometric titration), the Scripps Institution of Oceanography (manual titration), and the Woods Hole Oceanographic Institution (automated amperometric titration).

Differences in measured oxygen concentrations between institutions were encouragingly small. However, small, systematic differences in dissolved oxygen between institutions did exist. The range between the highest and lowest oxygen values reported by the 4 institutions never exceeded 0.6% over the entire concentration range studied (3.4 to 6.2 ml/l). The good agreement is probably due to the use of the essentials of Carpenter's (1965) modification of the Winkler method by all institutions.

The intercalibration revealed several aspects of dissolved oxygen measurements that require further research: (1) the intercalibration should be extended to very low oxygen concentrations; (2) procedures for measuring and applying corrections for the seawater blank need to be formalized; (3) a simple procedure to measure the temperature of seawater at the time of sampling needs to be developed; and (4) the solubility of atmospheric oxygen in the Winkler reagents must be measured as a function of temperature.

The intercalibration also revealed that analytical techniques required for precise and accurate volumetric measurements were often not applied, even by experienced analysts. It was found that uncalibrated pipets were used to dispense standards, that the volumes of oxygen flasks were not corrected for buoyancy, and that corrections for the thermal expansion of aqueous solutions were often not applied.

INTRODUCTION

This report describes an intercalibration of dissolved oxygen methods conducted between April 28 and May 3, 1990, during leg 3 of R/V Oceanus cruise 219. The experiment compared three techniques using automated endpoint detection with the manual Winkler method using a starch endpoint. Institutions participating in the intercomparison were the Bedford Institute of Oceanography (automated photometric titration), the University of Delaware (automated amperometric titration), the Scripps Institution of Oceanography (manual titration), and the Woods Hole Oceanographic Institution (automated amperometric titration).

The purpose of the intercalibration was twofold:

1) to determine the precision of each technique under seagoing conditions; and

2) to check each method for systematic errors relative to the classic manual Winkler titration using a starch endpoint.

The intercomparison was carried out at 2 stations in the Sargasso Sea (Figure 1 and Table 1). Vertical temperature, salinity, and oxygen profiles are shown in Figures 2 and 3. Station 2 (casts 21, 23, 24) is a repeat of KNORR 29 station 249 (9 July 1972), Geosecs station 121 (30 March 1973), and Transient Tracers station 005 (5 April 1981).

Table 1. Station summary for oxygen intercalibration, R/V Oceanus cruise 219.

Cast	Date	Time (GMT) Start	Time (GMT) End	Latitude (°N)	Longitude (°W)	Pressure Range Sampled (dB)	Water Depth (m)	Number of Depths Sampled
				Station 1				
13	29 April 90	07:25	08:36	33°46.78'	65°54.59'	0020 - 5195	5111	11
15	29 April 90	20:02	20:22	33°10.05'	65°44.97'	0896 - 0896	5111	10
16	29 April 90	22:19	23:09	33°49.09'	65°52.99'	3549 - 3550	5111	10
20	30 April 90	16:24	17:29	33°49.68'	65°53.85'	0022 - 5109	5122	10
				Station 2				
21	1 May 90	13:48	15:30	35°55.39'	67°50.47'	3040 - 4945	4926	10
23	1 May 90	23:30	23:50	35°55.97'	67°52.02'	0011 - 0909	4846	10
24	2 May 90	01:45	02:24	35°58.20'	67°55.02'	1014 - 2840	4950	10
				KNORR 29				
249	7 July 72			35°54'	67°56'		4931	
				Geosecs				
121	30 March 73			35°59'	67°59'		4933	
				Transient Tracers				
005	5 April 81			35°59'	68°00'		4928	

METHODS

Water Sampling

Seawater samples were collected with a General Oceanics rosette sampler equipped with 10 liter PVC Niskin bottles and a Neil Brown Mark III CTD. Samples were collected on the open deck, in an area with no overhead covering. At night the lighting was poor in this area, and, at certain positions around the rosette, it was impossible to check the oxygen drawing tubes for trapped air bubbles. The exposed sampling position became a problem after cast 24 when the ship was underway. As samples were drawn after this cast, the deck was often underwater to such an extent that cases containing the oxygen flasks were floating and in danger of being lost over the side. In addition, spray which had collected on the deck above dripped steadily onto the area in which the reagent dispensers were mounted; occasionally drops from the deck above would fall into an oxygen flask as it was being pickled. The digital thermometer used to measure sampling temperatures was flooded with seawater and damaged while cast 24 was being sampled.

Oxygen samples from Bedford, Delaware, and Scripps were collected in calibrated iodine flasks (Erlenmeyer flasks with a ground glass stopper and a lip to provide a water seal). Ground glass stoppers on the Bedford flasks contained long nipples which extended into the flask and displaced enough volume so that titrations did not overflow the flask (Green and Carritt, 1966). The Woods Hole samples were collected in calibrated narrow mouth amber reagent bottles with flat head ground glass stoppers (147.5 ml nominal volume).

Duplicate oxygen samples were drawn by each institution. The second of the two samples was drawn immediately after the first. The order in which samples were drawn was deliberately staggered, to detect possible systematic errors, as the head space in the Niskin bottles increased during sampling. The order in which samples were drawn from each Niskin bottle is listed in Table 2.

To minimize errors due to contamination from atmospheric oxygen, all oxygen samples from Niskin bottles 2-4 were drawn before the air vents on bottles 5-8 were opened. All samples from Niskin bottles 5-8 were then drawn before bottles 9-12 were opened. About 15 minutes was required to draw the 4 sets of duplicate oxygen samples from each Niskin bottle. About 45 minutes elapsed between the time the first and last samples were drawn from the rosette.

Oxygen samples were the first samples drawn from the Niskin bottles at station 1. At station 2, a freon sample (about 300 ml including rinse) was drawn before the first oxygen sample.

Oxygen flasks were copiously flushed with seawater before pickling reagents were added. The volume of seawater remaining in the Niskin bottles after all oxygen samples had been drawn was measured during cast 20. Between 1.5 and 3.0 liters of seawater remained in the 10 liter Niskin bottles. This indicates that about 1 liter of seawater was used to rinse and flush each

Table 2. Order of sampling for each Niskin bottle. Bottle 1 (and usually bottle 2) was used for other purposes during the cruise and was not sampled as part of the intercalibration.

Niskin bottle number	Relative position in cast	Order of sampling by institution			
		First	Second	Third	Fourth
2	deep	Bedford	Scripps	Delaware	Woods Hole
3		Woods Hole	Bedford	Scripps	Delaware
4		Delaware	Woods Hole	Bedford	Scripps
5		Scripps	Delaware	Woods Hole	Bedford
6		Bedford	Scripps	Delaware	Woods Hole
7		Woods Hole	Bedford	Scripps	Delaware
8		Delaware	Woods Hole	Bedford	Scripps
9		Scripps	Delaware	Woods Hole	Bedford
10		Bedford	Scripps	Delaware	Woods Hole
11		Woods Hole	Bedford	Scripps	Delaware
12	shallow	Delaware	Woods Hole	Bedford	Scripps

Table 3. Source of reagents used by each institution.

Institution	$MnCl_2$	NaOH/NaI	H_2SO_4
Woods Hole	Batch 1	Batch 1	Batch 1
Delaware	Batch 2	Batch 2	Batch 1
Scripps	Batch 2	Batch 2	Batch 2
Bedford	Batch 2	Batch 2	Batch 2

flask, since 8 oxygen flasks were filled from each Niskin bottle. The 1 liter seawater rinse corresponds to approximately 7-10 complete flushes of each oxygen flask. This degree of flushing is much greater than the 1/3 bottle volume recommended by Strickland and Parsons (1965), the 1 bottle volume by Carpenter (1965), and the 2 bottle volumes by Horibe et al (1972) and by Grasshoff (1976).

It is our opinion that the use of proper water sampling techniques is the single most important factor in obtaining high quality oxygen measurements.

Reagent Addition

Reagents used to pickle and acidify the samples were prepared at Woods Hole, using concentrations specified by Carpenter (1965). Two liters of each reagent were prepared and filtered through a glass fiber filter (Gelman type A/E, 1.6 μm pore size, or Whatman type GF-F, 0.7 μm pore size) to remove particulates. The reagents were dispensed with all glass and teflon Dispensette Bottle-Top Dispensers, 0-2 ml capacity, from Brinkmann Instruments. The volume of each dispenser was adjusted to 1 ml by dispensing 10 portions into a 10 ml graduated cylinder.

The length of the tips of the $MnCl_2$ and NaOH/NaI reagent dispensers used by Bedford, Delaware, and Scripps was increased from 1 to 3 inches by an additional length of small diameter plastic tubing. The tips were lengthened to ensure that manganese hydroxide did not precipitate in the necks of the oxygen flasks before they were stoppered. Erroneously high oxygen concentrations would be calculated if oxidized precipitate from the neck of the flask settled into the interior of the flask before it was stoppered.

Because the analysts were located in 2 different locations aboard ship, it was necessary to divide the reagents into 2 batches. Table 3 lists the batch of reagents used by each institution. Reagent blanks for both sets of reagents were measured by each institution.

After copious flushing, the filled oxygen flasks were carried to a nearby rack containing the $MnCl_2$ and NaOH/NaI pickling reagents, which were immediately injected into the samples. The flasks were stoppered and then inverted and shaken to mix the reagents. The flasks were not stoppered during the short interval between sampling and reagent addition.

The pickled oxygen samples were returned to the laboratory; once the precipitate had settled, the flasks were shaken a second time, and the precipitate allowed to settle before titration.

Analytical Methods

Details of the methods used by each of the four institutions are listed in Table 4. **All standards and blanks were prepared and run in distilled water.** All institutions used Carpenter's (1965) method of reverse 1 ml reagent additions to determine the reagent blank.

Table 4. Details of oxygen methods used on R/V Oceanus Cruise 219

	Bedford	Delaware	Scripps	Woods Hole
Endpoint:	photometric	amperometric	starch	amperometric
Volumes:				
Flask volume (ml)	131	145	104	147.5
Aliquot volume (ml)	-	-	-	49.858
Size of buret (ml)	5	5	1	10
Smallest measurable volume increment (μl)	1	1	0.1	1
Reagent concentrations:				
$MnCl_2$	3 molar			
NaOH/NaI	8 molar & 4 molar			
H_2SO_4	10 normal			
KIO_3 or $KH(IO_3)_2$	0.01 normal			
KIO_3 volume (ml)	9.9656	9.9784	10	15
Thiosulfate normality	0.057	0.141	0.141	0.0101
$MnCl_2$ in standards	no	no	yes	yes
Wetting agent added	yes	no	no	no

Bedford

The automated titration used by the Bedford Institute of Oceanography was a modification of the manual Winkler titration previously employed at Bedford (Levy et al, 1977a,b). The automated titration differed from Carpenter's (1965) method in four respects: (1) the starch endpoint was replaced by a photometric endpoint; (2) the 1 ml manual microburet was replaced by a 5 ml Metrohm Dosimat E655 digital piston buret; (3) the thiosulfate normality was reduced from 0.14 to 0.06; and (4) 1 drop of wetting agent was added to all samples, standards, and blanks just prior to titration to prevent bubble formation.

The photometric endpoint was detected at a wavelength of 420 nm with a Brinkmann model PC 800 digital fiber optic probe colorimeter. The titration program was written in Pascal; the titration was controlled by an IBM compatible microcomputer.

Standards were dispensed with a 10 ml, class A, calibrated hand pipet; blanks with a 1 ml, class A, hand pipet. The standard KIO_3 solution was a commercially prepared 0.01 normal solution (BDH R02592, lot 9001142) distributed in 100 ml polyethylene bottles. A freshly opened bottle was used for each standardization.

The Bedford automatic titrator developed erratic behavior after cast 20, apparently due to an incompatibility between the microcomputer and the ship's power supply. Consequently, only Bedford data from casts 13 through 20 are included in this report.

Delaware

The automated titration used by the University of Delaware differed from Carpenter's (1965) method in two respects: (1) the starch endpoint was replaced by an amperometric endpoint; and (2) the 1 ml manual microburet was replaced by a 5 ml Metrohm Dosimat E535 digital piston buret.

The titration program was written in Commodore Basic; the titration was controlled by a Commodore CBM 8032 microcomputer using the procedure described by Culberson and Huang (1987). The average analysis time per sample, including acidification, sample changing, and titration was 4.8 minutes.

Standards were dispensed with a 10 ml, class A, calibrated hand pipet; blanks with a 1 ml Eppendorf pipet. The standard KIO_3 solution was a 2 liter batch of KIO_3 prepared on 11 April 1990. Fisher reagent grade KIO_3 (P253-100, lot 897675, assay 99.9%) was dried at 135°C for 12 hours, cooled in a desiccator, and weighed to 0.1 mg. The weighed salt was transferred to a 2 liter, class A, volumetric flask and made up to volume at 22.5°C. The calculated normality was 0.010140. The weight of KIO_3 was corrected for buoyancy assuming a density of 3.89 (Hodgman, 1958); the molecular weight of KIO_3 was taken as 214.001.

Scripps

The manual titration with starch endpoint used by Scripps (Dickson and Anderson, 1990) was identical to that described by Carpenter (1965) with one exception. In addition to the 1 ml of H_2SO_4 and 1 ml of NaOH/NaI specified by Carpenter (1965), Scripps also added 1 ml of $MnCl_2$ to their standards. The average analysis time per sample, including acidification, sample changing, and titration was 4.0 minutes.

Standards were dispensed with an uncalibrated 10 ml automatic pipet; blanks with a 1 ml Eppendorf pipet. The standard iodate solution was prepared, during the cruise, in 1 liter batches from pre-weighed (to 0.1 mg) vials of Fisher reagent grade KIO_3 (P253-100, lot 870977, assay 100.2%). The normality of the standard was assumed to be exactly 0.010000. Two 1 liter batches of standard were prepared during the cruise, their thiosulfate titers differed by 0.04%.

The 10 ml automatic pipet used to dispense standards had not been calibrated prior to the cruise, and was broken, after the cruise, before it could be calibrated. The volumes of 8 similar pipets, previously calibrated at Scripps, ranged from 9.9957 to 9.9726 ml, and averaged 9.985 ± 0.007(1σ) ml.

Woods Hole

The automated titration used by Woods Hole Oceanographic Institution (Knapp, Stalcup, and Stanley, 1989, 1990) differed from Carpenter's (1965) method in several respects: (1) an aliquot of the acidified sample, rather than the entire contents of the sample bottle, was titrated; (2) the starch endpoint was replaced by an amperometric endpoint; (3) the 1 ml manual microburet was replaced by a 10 ml Metrohm Dosimat E665 digital piston buret; (4) the thiosulfate normality was reduced from 0.14 to 0.01 normal; and (5) 1 ml of the $MnCl_2$ reagent was added to their standards.

In the Woods Hole procedure, a 50 ml aliquot of the acidified sample is added to a 100 ml beaker and the contents titrated with 0.01 normal thiosulfate. The average analysis time per sample, including acidification, sample changing, and titration was 2.5 minutes.

The standardization procedure used by Woods Hole differs from that of Carpenter (1965). In the Woods Hole procedure, 150 ml of distilled water was added to a 200 ml Erlenmeyer flask; 1 ml each of the H_2SO_4, $MnCl_2$, and NaOH/NaI reagents were then added with stirring; and finally, 15 ml of the iodate standard was added to the flask. A 50 ml aliquot of this solution was titrated as if it were an unknown. This procedure was adopted to minimize errors due to iodine volatilization.

Standards and blanks were dispensed with a 20 ml Metrohm Dosimat E665 digital piston buret. The standard iodate solution was a 1 liter batch of $KH(IO_3)_2$ prepared on 20 March 1990. Fisher purified $KH(IO_3)_2$ (P190-100, lot 893220, assay 100.1%) was dried in a desiccator and weighed to 0.1 mg. The weighed salt was transferred to a 1 liter, class A, volumetric flask and made up to volume with distilled water.

CALCULATIONS

The following equations were used to calculate the concentration of dissolved oxygen in the seawater samples. All volumes are expressed in milliliters.

V_x = thiosulfate titer of sample (ml)

$V_{blk,dw}$ = thiosulfate titer of pure water blank (ml)

V_{std} = thiosulfate titer of standard (ml)

V_{bot} = volume of sample bottle (ml)

V_{aliq} = volume of aliquot (50 ml)

V_{reg} = volume (2 ml) of sample displaced by reagents

V_3 = volume of reagents (3 ml) used to prepare standards in aliquot method

V_{150} = volume of distilled water (150 ml) used to prepare standards in aliquot method

V_{IO3} = volume of iodate standard (ml)

N_{IO3} = normality of iodate standard (= 6·molarity)

DO_{reg} = absolute amount of oxygen added with reagents, 0.0017 ml (Murray, Riley, and Wilson, 1968)

O_2 = oxygen concentration in sample (ml/l)

Whole Bottle Titration

Dissolved oxygen calculations for whole bottle titrations performed by Bedford, Delaware, and Scripps were calculated from the following modification of Carpenter's (1965) equation,

$$O_2 = \frac{\dfrac{(V_x - V_{blk,dw}) \cdot V_{IO3} \cdot N_{IO3} \cdot 5598}{(V_{std} - V_{blk,dw})} - 1000 \cdot DO_{reg}}{(V_{bot} - V_{reg})} \qquad (1)$$

Equation 1 differs from Carpenter's (1965) equation for whole bottle titrations in the correction for oxygen added with the reagents. In equation 1, the correction for oxygen added with the reagents is a function of the flask volume; whereas, in Carpenter's equation, a constant correction, 0.018 ml/l, is used. The use of a constant value for the concentration of oxygen added with the reagents is only correct if all flasks have identical volumes.

Titration of an Aliquot

The normality of thiosulfate used to titrate the Woods Hole samples was calculated from equation 2,

$$N_{thio} = \frac{V_{IO3} \cdot N_{IO3}}{\left(V_{std} \cdot \frac{(V_{150} + V_{IO3} + V_3)}{V_{aliq}} - V_{blk,dw}\right)} \qquad (2)$$

Dissolved oxygen calculations for titrations performed by Woods Hole were calculated from equation 3,

$$O_2 = \frac{\left(V_x \cdot \frac{V_{bot}}{V_{aliq}} - V_{blk,dw}\right) \cdot N_{thio} \cdot 5598 - 1000 \cdot DO_{reg}}{(V_{bot} - V_{reg})} \qquad (3)$$

RESULTS

Measurement of dissolved oxygen concentration involves several steps: (1) water sampling; (2) addition of reagents; (3) standardization of the thiosulfate titrant; (4) determination of the reagent and sample blanks; and (5) titration of the seawater sample. Items (1) and (2) were discussed under METHODS. The last 3 items represent the titration of samples, standards, and blanks and will be discussed in this section.

Thiosulfate Standardization

The standards run by each institution were prepared by dispensing a precise, known volume of iodate into a ; adding the H_2SO_4 and NaOH/NaI (and sometimes $MnCl_2$) reagents; and titrating the liberated iodine with thiosulfate. Statistical analysis of replicate standards provides a measure of the best possible precision of a particular analytical technique. The actual precision of oxygen analyses may be worse than the precision calculated from replicate standards, due to the presence of sampling errors which occur when oxygen samples are drawn.

The average thiosulfate titer and its associated standard deviation, for each set of standards run by each institution, is shown in Figures 4 through 7. A statistical analysis of this data is given in Table 5.

Table 5. Precision of replicate standards run by different institutions during R/V Oceanus cruise 219. Standard deviation calculated by pooling the variances of the individual sets of standards (Youden, 1951). This table contains some data not shown in Figures 4 through 7.

Institution	Sets of replicate standards	Number of replicates per set	Relative standard deviation
Bedford	6	5	0.15%
Delaware	10	5	0.09%
Scripps	8	4	0.06%
Woods Hole	9	4	0.06%

An examination of the results in Table 5 and Figures 4 through 7, shows that standards run by Woods Hole and Scripps are considerably more precise than those of Bedford and Delaware. The difference between the precisions of the Woods Hole and Delaware standards, both of which use Metrohm Dosimat titrators and amperometric endpoints, may be due to the non-diffusive buret tip used by Woods Hole, but not by Delaware. The one-way valve in this buret tip eliminates diffusion of thiosulfate into the solution between additions of titrant. On a subsequent cruise in which a non-diffusive buret tip was used, the relative standard deviation of Delaware standards was 0.07% for 9 sets of 5 replicates measured over a 2 week period.

The normality of the standard used by each institution was checked after cast 24. The thiosulfate titer of five replicates of each standard was measured by the automated amperometric titration used by Delaware (Table 6). Although this experiment cannot yield the absolute normality of each standard, it does provide information on their relative concentrations.

Table 6. Comparison of standards from different institutions. Five replicates of each standard were titrated with the Delaware autotitrator. The t-statistic was calculated from the overall standard deviation for the Delaware standards, 0.00065 (= 0.09%), given in Table 5.

Institution	Nominal normality	Average titer	Standard deviation	Ratio to Woods Hole	t statistic
Bedford	0.01000	0.7046_6	0.0003	0.99497	12.23
Scripps	0.01000	0.7083_4	0.0009	1.00017	0.41
Woods Hole	0.01000	0.7082_2	0.0007	1.00000	--
Delaware	0.01014 (0.71762/1.014 =)	0.7176_2 0.7077_1	0.0006	0.99928	1.75

Since the nominal normalities of the Bedford, Scripps, and Woods Hole standards were all 0.01, there should be no difference between the average titers listed in Table 6. The 0.02% difference between the titers of the Scripps and Woods Hole standards is not significant (P = 69%). Furthermore, the 0.07% difference in titer between the Delaware (normalized to 0.01) and Woods Hole standards is not significant (P = 12%). However, the 0.5% difference between the Bedford and Woods Hole titers is highly significant (P < 0.5%).

Since the normalities of the Delaware, Scripps, and Woods Hole standards are consistent with each other, it appears that the true normality of the Bedford standard is lower than the nominal value. The correct normality of the Bedford standard, calculated from the average titer of the Delaware, Scripps, and Woods Hole standards ([0.7077 + 0.7083 + 0.7082]/3 = 0.7081), is 0.009952 (= 0.01*0.7047/0.7081). **The oxygen concentrations listed for Bedford in this report are calculated using an iodate normality of 0.009952.**

Blank Determination

Three factors may contribute to the blank measured by Carpenter's (1965) procedure of successive 1 ml KIO_3 additions:

1) any difference between the measured end-point and the equivalence point, $V_{blk,ep}$;
2) the presence of oxidants or reductants in the reagents, $V_{blk,reg}$; and
3) the presence of oxidants or reductants (other than oxygen) in the sample, $V_{blk,x}$.

Blanks measured during this intercalibration were determined in deionized water. These pure water blanks are the sum of factors 1 and 2 above; they measure the concentration of redox species in the pickling reagents plus any bias in the measured end-point.

$$V_{blk,dw} = V_{blk,ep} + V_{blk,reg}. \qquad (4)$$

Blanks determined in pure water do not measure the concentration of oxidants or reductants in the seawater samples.

For a given method, the end-point, $V_{blk,ep}$, and reagent, $V_{blk,reg}$, blanks are constants independent of the sample volume. However, the sample blank, $V_{blk,x}$, is a function of the sample volume, since it reflects actual concentrations of redox species in the sample.

Seawater blanks were not measured during the intercalibration. In hindsight, this was a mistake.

A practical difficulty in using seawater blanks, is that, rigorously, sample blanks should be measured for each seawater sample, to allow for variation in the concentrations of oxidants and reductants.

Each of the institutions participating in the intercalibration, determined the pure water blank using Carpenter's (1965) procedure. The blanks measured by each institution cannot be directly compared because the normality of thiosulfate differed between institutions. Consequently in Table 7, we have converted the blank measured by each institution into the equivalent amount of dissolved oxygen using the following modification of equation 1.

$$O_{2,blk,dw} = \frac{V_{blk,dw} \cdot N_{thio} \cdot 5598}{125} \qquad (5)$$

The oxygen concentration corresponding to a given blank is calculated for a nominal volume of 125 ml.

Table 7. Comparison of pure water blanks measured by different institutions on 2 May. Blanks calculated from equation 5, and expressed as oxygen concentrations in ml/l.

	Woods Hole Reagents (batch 1)			
	Bedford	Delaware	Scripps	Woods Hole
Average blank (ml/l)	0.0282	0.0088	-0.0088	0.0056
Std deviation (ml/l)	0.0050	0.0019	0.0025	0.0013
	Bedford/Scripps Reagents (batch 2)			
Average blank (ml/l)	0.0332	0.0006	-0.0044	0.0050
Std deviation (ml/l)	0.0132	0.0025	0.0025	0.0013

Several features are evident in Table 7. First, blanks determined by Bedford are considerably larger than those of the other institutions. Second, Delaware and Woods Hole blanks are roughly equal. Third, Scripps blanks are slightly less than the Delaware and Woods Hole blanks. Scripps blanks being more negative than Delaware and Woods Hole blanks is consistent with the fact that the starch endpoint occurs slightly before the amperometric endpoint (Bradbury and Hambly, 1952; Knowles and Lowden, 1953; Culberson and Huang, 1987).

Oxygen Measurements

A complete list of the oxygen concentrations measured by each institution is given in Appendices A through E.

Precision of Individual Methods

The precision of oxygen measurements from each institution was calculated by pooling differences between duplicates (Youden, 1951, page 16). The data from each institution is highly precise, with standard deviations ranging from 0.005 to 0.010 ml/l (Table 8). If the few duplicates with differences > 0.05 ml/l (10 standard deviations of the most precise method) are excluded, the precision of the Delaware, Scripps, and Woods Hole techniques are all between 0.004 and 0.005 ml/l.

Table 8. Precision (1 standard deviation) of oxygen measurements from different institutions, as measured by the difference between the oxygen concentrations of duplicate samples.

	Bedford	Delaware	Scripps	Woods Hole
	calculated using all measured values			
Precision (ml/l):	0.010	0.005	0.007	0.008
Number of duplicates:	35	67	70	70
	using duplicates with differences < 0.05 ml/l			
Precision (ml/l):	0.010	0.005	0.004	0.005
Number of duplicates:	35	67	69	69

Plots of the difference between duplicates versus the oxygen concentration (Figures 8 - 11) indicate that the precision of each method is independent of concentration over the range of concentrations measured.

For each institution, the order in which oxygen samples were titrated matched the order in which they were drawn. If the measured concentrations were independent of the order in which the samples were drawn, the differences between duplicates should be symmetrical about the origin. Histograms (Figures 12 - 15) show that this is true.

Systematic Differences between Institutions

Comparison of the oxygen concentrations in Appendix A shows that there are small systematic differences between the concentrations reported by different institutions. The average difference between oxygen concentrations reported by each pair of institutions is summarized in Table 9.

Table 9. Average difference (ml/l) between oxygen concentrations reported by pairs of institutions (institution A minus institution B). The average difference was calculated by summing the individual differences and dividing by the total number of samples. The number of common samples is given in parenthesis after each difference.

Institution B	Institution A			
	Bedford	Delaware	Scripps	Woods Hole
Bedford	------	-0.010 (35)	0.013 (35)	-0.007 (35)
Delaware		------	0.020 (71)	0.002 (71)
Scripps			------	-0.019 (71)
Woods Hole				------

Close examination of the systematic differences between methods (Figures 16 - 21) shows that they are not constant, but tend to increase with increasing oxygen concentration. With one exception (Table 10), the slopes and intercepts of the regression lines plotted in Figures 16 through 21 are significantly different from zero at a 99% confidence interval.

To facilitate comparison of the systematic differences between institutions, regression lines from Figures 16 through 21 are plotted on Figure 22.

The fact that the bias between institutions increases with increasing concentration (regression lines in Figure 22 have positive slopes) could be due to small errors in constant factors, such as V_{103} and N_{103}, used in equations 1 and 3.

Table 10. Regression analysis of systematic differences between oxygen concentrations (ml/l) reported by different institutions. Rows labeled Probability are the probability of the intercept or slope equaling zero.

	Institutions					
	DEL-BIO	SIO-BIO	WHOI-BIO	SIO-DEL	SIO-WHOI	WHOI-DEL
Intercept:	-0.0354	-0.0415	-0.0510	-0.0052	0.0098	-0.0149
Standard deviation:	0.0054	0.0050	0.0058	0.0031	0.0034	0.0037
t-statistic:	6.61	8.27	8.75	1.70	2.92	4.05
Probability:	<0.5%	<0.5%	<0.5%	<10%	<1%	<0.5%
Slope:	0.0054	0.0113	0.0092	0.0050	0.0017	0.0032
Standard deviation:	0.0011	0.0010	0.0012	0.0006	0.0006	0.0007
t-statistic:	4.98	11.12	7.77	8.48	2.71	4.59
Probability:	<0.5%	<0.5%	<0.5%	<0.5%	<1%	<0.5%

Systematic differences between methods are unlikely due to errors in N_{IO3}, since the normalities of the iodate standards used in the oxygen calculations were intercompared (Table 6) and adjusted where necessary.

However, as mentioned in the Methods section, the automatic 10 ml pipet used by Scripps to dispense iodate standards was broken before it could be calibrated. Scripps oxygen concentrations in this report have been calculated assuming a volume (V_{IO3}) of exactly 10 ml for the iodate pipet. A -0.3% error in the assumed volume of the Scripps pipet would account for the difference in reported oxygen concentrations between Scripps and Delaware/Woods Hole (Table 9). The volumes of 8 similar 10 ml pipets, previously calibrated by Scripps (see Methods section), averaged -0.15% low. Consequently, it is possible that much of the difference between the Scripps and Delaware/Woods Hole oxygen concentrations is due to the volume of the Scripps pipet being less than the assumed 10 ml.

For comparison, the volumes of the 10 ml hand pipets used by Bedford and Delaware to dispense iodate standards (Table 4) were 0.34% and 0.22% too low, respectively.

The non-zero intercepts shown in Figures 16 through 21 may be due to differences between the starch, amperometric, and colorimetric end-points, and the true equivalence point in the titration of iodine with thiosulfate.

Effect of Drawing Order on Oxygen Concentrations

Since 70-90% of the seawater in each 10 liter Niskin bottle was used during sampling, it is possible that the last samples collected from the Niskin bottles were contaminated with atmospheric oxygen. The order in which institutions sampled each Niskin bottle was deliberately staggered to detect atmospheric contamination (see Table 2).

The statistical analysis of sampling order in Table 11 indicates that there was no detectable difference between oxygen concentrations of the first and last samples drawn.

Conversion of Volumetric Concentrations to Weight Concentrations

The analytical methods used to determine dissolved oxygen during this intercalibration were volumetric techniques. They determine the amount of dissolved oxygen per unit volume of seawater.

Current oceanographic practice requires data to be reported as weight concentrations, on a per kilogram of seawater basis. Dividing the volumetric concentration by the density of seawater yields the weight concentration. Since oxygen samples are pickled immediately after being drawn, the density of seawater used in the conversion should be calculated at the temperature of the seawater at the time of sampling. This temperature is not routinely measured. Instead, it is assumed that the water samples do not warm as they are brought onboard, and the density is calculated at the potential temperature of the

Table 11. Effect of drawing order on measured oxygen concentration. The column labeled Number of Samples is the number of samples available for comparison. The column labeled Average Oxygen is the oxygen concentration averaged over the given number of samples. The column labeled Average Difference is the average difference in oxygen concentrations reported by the two institutions, for the given number of samples. If the last samples drawn are subject to atmospheric contamination, the Average Difference for the first row of the comparison should be significantly different than the Average Difference for the second row of the comparison.

--------- Sampling Order ----------	Number of Samples	Average Oxygen (ml/l)	Average Difference (ml/l)	Standard Deviation of Differences (ml/l)
Rosette positions 3, 7, 11; Woods Hole first, Delaware last:	21	5.055	0.002	0.009
Rosette positions 4, 8, 12; Delaware first, Woods Hole second:	21	5.044	0.000	0.005
		Combined standard deviation for both sets:		0.008
		t-statistic for difference between positions 3,7,11 and 4,8,12:		0.84
Rosette positions 4, 8, 12; Delaware first, Scripps last:	21	5.063	0.019	0.006
Rosette positions 5, 9; Scripps first, Delaware second:	14	5.124	0.020	0.007
		Combined standard deviation for both sets:		0.007
		t-statistic for difference between positions 4,8,12 and 5,9:		0.44

sample. If the sampling and potential temperatures are different, weight concentrations calculated from the potential density will be in error, by as much as 0.5% for a 25°C difference (Kester, 1975).

To assess the difference between the sampling and potential temperatures, the sampling temperature was determined at several stations by R. T. Williams. Temperatures were measured with a platinum resistance thermometer attached to a digital thermometer readable to 0.1°C. The platinum resistance thermometer was embedded in a short piece of plastic tubing which fit over the Niskin bottle outlet. Seawater temperatures in the Niskin bottles were measured immediately before the first oxygen sample was drawn, and immediately after the last sample was drawn. The air temperature was 20 ± 1°C during these experiments.

Figure 23 shows the experimental results. The coldest seawater had warmed 2 to 6°C by the time of first sampling; it had warmed 5 to 10°C by the time the last sample was drawn. Sampling each Niskin bottle took approximately 15 minutes. Since only 1 set of oxygen samples are normally drawn, the temperature changes occurring at the time of first sampling are probably most representative of temperature differences that would be encountered in routine measurements. Extrapolating these results to locations with significantly different vertical temperature profiles and air temperatures is not recommended.

The change in seawater temperature between the beginning of sampling and the end of sampling is shown in Figure 24. The coldest seawater warmed about 4°C in the 15 minutes required to draw all oxygen samples from a single Niskin bottle.

The maximum 4°C temperature increase in Figure 24, represents a 0.06% decrease in the seawater density. Theoretically, this density decrease during the course of sampling would cause the volumetric oxygen concentration of the last sample drawn to be 0.06% lower than that of the first sample drawn. For North Atlantic Deep Water with oxygen concentrations of 6 ml/l, the theoretical change in the volumetric oxygen concentration would be 0.004 ml/l.

The results of the oxygen intercalibration have not been converted from volumetric to weight concentrations for the following reasons:

1) systematic errors caused by neglecting temperature changes during sampling are small (<0.06%); and

2) sampling temperatures were not measured for every station and/or sample.

DISCUSSION

Systematic differences in dissolved oxygen (Figure 22) between the institutions participating in the intercalibration were encouragingly small. The largest difference, that between Woods Hole and Bedford at low concentrations, was -0.6%. All other differences were within ±0.5% over the concentration range studied (3.4 to 6.2 ml/l). Relative differences between institutions at the lowest and highest oxygen concentrations measured are listed in Table 12.

Table 12. Systematic differences in dissolved oxygen between institutions calculated from the regression lines in Figures 16 through 21. The listed values are the percent difference in dissolved oxygen between institutions for a given oxygen concentration.

--- Institutions ----	Percent difference at 3.4 ml/l	6.2 ml/l
Delaware - Bedford	-0.5%	0.0%
Scripps - Bedford	-0.1%	0.5%
Woods Hole - Bedford	-0.6%	0.1%
Scripps - Delaware	0.3%	0.4%
Woods Hole - Delaware	-0.1%	0.1%
Scripps - Woods Hole	0.5%	0.3%

Systematic differences between Scripps and Delaware/Woods Hole were approximately constant at 0.3% to 0.5% over the entire concentration range studied. Differences between Woods Hole and Delaware were much less; averaging only 0.002 ml/l (Table 9).

As mentioned above, Scripps results higher than those of Delaware and Woods Hole may be due to the uncalibrated pipet used to deliver the Scripps standard.

The good agreement between methods is probably due to the use of the essentials of Carpenter's (1965) method by all institutions. In a previous intercomparison between 11 institutions (Carritt and Carpenter, 1966) before adoption of Carpenter's (1965) technique, the range of concentrations reported for air saturated seawater was 9%.

The oxygen concentrations measured during the intercalibration varied between 53% and 104% saturation (Figures 2 and 3). Consequently, it was not possible to intercalibrate at low concentrations where errors due to sampling, blanks, and oxygen in the reagents will be most important. However, the non-zero intercepts of the regression lines in Figure 22 suggest that systematic errors between some of the methods may increase at low concentrations. The extrapolated values at zero oxygen are particularly large for differences involving the Bedford Institute of Oceanography.

Because the precision (σ <= 0.010 ml/l) of the present methods is high, other sources of error in equations 1 and 3 become important, particularly at low oxygen concentrations. Two potential sources of error are (1) the concentration of dissolved oxygen in the reagents and (2) the seawater blank.

Oxygen in the Reagents

For a nominal oxygen volume of 125 ml, the amount of oxygen added with the reagents is 0.014 ml/l (= 0.0017/0.123). This value is 3 times the standard deviation of the most precise of the present methods. Variation of the amount of oxygen added with the reagents is, potentially, a major source of error in calculated oxygen concentrations. The amount of oxygen dissolved in the reagents, 0.0017 ml, was determined at 25.5°C (Murray et al, 1968). Reagents stored at lower temperatures will presumably contain more oxygen. Ideally, the $MnCl_2$ and NaOH/NaI reagents should be stripped of oxygen and stored under an oxygen free atmosphere. Since this does not appear feasible under shipboard conditions, we recommend that the reagents be stored in the ship's laboratory and not brought on deck until needed. This procedure should minimize large changes in the concentration of oxygen in the reagents.

The reagents used during the intercalibration were divided into 2 batches since the analysts were in different laboratories aboard ship. It is possible that the concentration of oxygen in the 2 batches of reagents differed slightly due to small differences in storage temperature (22 ± 3°C).

The Seawater Blank

A potential source of error in equations 1 and 3 is the value of the seawater contribution to the blank. During the course of the intercalibration it became apparent that there was almost no information available on the magnitude and variability of seawater blanks.

Pure water ($V_{blk,dw}$) and seawater blanks ($V_{blk,sw}$) can be represented by equations 4 and 6,

$$V_{blk,dw} = V_{blk,ep} + V_{blk,reg} \quad (4)$$

$$V_{blk,sw} = V_{blk,x} + V_{blk,ep} + V_{blk,reg} \quad (6)$$

where

$V_{blk,ep}$ = blank due to differences between the measured end-point and the equivalence point;

$V_{blk,reg}$ = blank due to oxidants or reductants in the reagents; and

$V_{blk,x}$ = blank due to presence of oxidants or reductants (other than oxygen) in the sample.

Rigorously, it is the seawater blank ($V_{blk,sw}$) which should be subtracted from V_x, the thiosulfate titer of the sample, in equations 1 and 3.

In practice, blanks used in equations 1 and 3 have been estimated by two different approaches.

1) Seawater and pure water blanks are assumed equal, and the pure water blank is used throughout equations 1 and 3. This approach is used by Bedford, Delaware, Woods Hole, and sometimes Scripps, and was adopted for the intercalibration.

2) The seawater blank is assumed constant, independent of position, and a single batch of seawater is used to prepare all standards and blanks. This procedure has been used by Scripps.

For a given oxygen method, the end-point ($V_{blk,ep}$) and reagent ($V_{blk,reg}$) blanks are constants independent of the sample volume. However, the sample blank ($V_{blk,x}$) is a function of the sample volume, since it reflects actual concentrations of redox species in seawater.

If we assume that the end-point blank ($V_{blk,ep}$) is identical in pure water and in seawater, the difference in blanks or standards determined in seawater and in water is,

$$V_{blk,sw} - V_{blk,dw} = V_{blk,x}. \qquad (7)$$

We could find only 13 measurements from which the sample blank ($V_{blk,x}$) could be calculated (Table 13). Iodate is probably one of the major contributors to the seawater blank, and values of $O_{2,blk}$ corresponding to various iodate concentrations are included in Table 13. Seawater of 35 ppt contains approximately 450 nanomolar iodate (Wong and Brewer, 1974). Iodate is not conservative and 30 ppt coastal seawater may contain as little as 200 nanomolar iodate. Based on the few available measurements, it appears that the sample blank in the open ocean, Sargasso Sea, and standard seawaters analyzed by Scripps and Woods Hole is close to the seawater iodate blank. Sample blanks in the 2 coastal samples analyzed by Woods Hole and Delaware are less than the iodate blank.

The range between the highest and lowest seawater blanks, 0.03 ml/l (Table 13), amounts to 1% of an oxygen concentration of 3 ml/l and 0.5% of a concentration of 6 ml/l. Clearly, failure to apply the proper seawater blank in equations 1 and 3 may cause errors in the calculated concentration much larger than those due to analytical imprecision.

Table 13. Measurements of the sample blank ($V_{blk,x}$) in seawater. Since the blanks measured by different institutions used differing seawater volumes and thiosulfate normalities, measured values have been converted to an equivalent concentration of oxygen using the following equation,

$$O_{2,blk} = \frac{V_{blk,x} \cdot N_{thio} \cdot 5598}{V_{seawater}} \qquad (8)$$

In some cases, $V_{blk,x}$ was calculated from the difference in volumes of standards determined in seawater and in pure water.

Date	Institution	Source of Seawater	Calculated from	$O_{2,blk}$ (ml/l)
07/01/81	Scripps	Open ocean	Standard	0.015
			Standard	0.013
12/06/87			Blank	0.010
			Standard	0.015
02/16/88			Standard	0.011
06/15/88			Standard	0.018
			Standard	0.015
05/14/90	Woods Hole	Standard seawater	Blank	0.010
			Standard	0.012
05/30/90			Blank	0.011
		Sargasso seawater	Blank	0.011
		Woods Hole harbor	Blank	0.000
06/30/90	Delaware	Coastal, 35.0 ppt, IO_3 = 484 nM	Blank	-0.011
		——— Calculated Iodate Blank ———		
		200 nanomolar IO_3		0.007
		300 nanomolar		0.010
		400 nanomolar		0.013
		500 nanomolar		0.017

RECOMMENDATIONS

Problems Needing Further Research

Based on the results of this intercalibration, we feel that the following items require additional study before methods can be guaranteed accurate to ±0.5% over the entire oceanic range of oxygen concentrations.

Low oxygen intercalibration

The intercalibration of oxygen methods should be extended to include very low oxygen concentrations. Intercalibration at low concentrations could be performed in the laboratory (Carritt and Carpenter, 1966), or at sea off the West Coast of the United States. Intercalibration at low concentrations will provide a severe test of sampling techniques.

The intercomparison should include a sample of oxygen free water prepared by purging the sample with gas free of oxygen. Measurements on oxygen free water will serve to check the values for oxygen in the reagents and for the blank used in equations 1 and 3.

Endpoint bias in pure and sea waters

The starch endpoint occurs slightly before the amperometric endpoint (Bradbury and Hambly, 1952; Knowles and Lowden, 1953). There is some evidence (Culberson and Huang, 1987) that the difference between the two endpoints varies with salinity. If the difference between the starch and amperometric endpoints does depend on salinity, there will be a systematic difference between the two methods when standards are run in pure water, and the samples are seawater.

The effect of salinity on endpoint bias between the starch, amperometric, and photometric endpoints should be studied. The simplest way to determine endpoint bias would be to determine standards and blanks in pure water and in seawater, using the same reagents for each analytical method. This study could be performed during the low oxygen intercalibration.

Seawater blanks

A systematic study of the difference between seawater and pure water blanks should be undertaken using Carpenter's (1965) blank procedure. The study should include seawater from both coastal and open ocean locations. Depths sampled should include the surface, the oxygen minimum, the nutrient maximum, and the bottom.

To increase precision, dilute thiosulfate (0.01 N) should be used in this study, since blanks determined with 0.14 N thiosulfate (Carpenter, 1965) are small and near the limit of detection.

Temperature warming in water bottles

The measurements of sampling temperature begun during this intercalibration should be extended to other areas of the ocean, and to other seasons. Measurements in the tropics, an area with high (30°C) air and surface seawater temperatures, are particularly important. The sampling temperature should be measured just prior to drawing the oxygen sample. Additional information required are the air temperature, the time of sampling, and the time the water bottles were brought on deck. This information would allow estimation of the error involved in conversion of volumetric to weight concentrations.

If possible, a simple technique for determining and recording the sampling temperature, that does not require operator assistance, should be devised.

Oxygen dissolved in the reagents

The solubility of atmospheric oxygen in the $MnCl_2$ and NaOH/NaI reagents should be measured (or at least calculated) as a function of temperature. If possible a technique for storing the reagents free of oxygen should be devised.

Techniques for Routine Oxygen Measurements

The following procedures should be made mandatory for all WOCE oxygen measurements. Although these are standard procedures for high precision measurements, our experience is that many oceanographic laboratories do not implement all of them.

Sampling procedure

All oxygen samples must be flushed with at least 2 volumes of seawater. The $MnCl_2$ and NaOH/NaI reagents should be added immediately after the sample is drawn, and before the oxygen is stoppered. Stoppering the oxygen s before adding the pickling reagents increases the chance of contamination with atmospheric oxygen.

The $MnCl_2$, NaOH/NaI, and H_2SO_4 reagents must have the concentrations specified by Carpenter (1965). Use of these reagents will minimize uncertainties due to the amount of oxygen added with the reagents.

It is our opinion that the use of proper water sampling techniques is the single most important factor in obtaining high quality oxygen measurements.

Calculation of dissolved oxygen

Equations 1 or 3 must be used to calculate the concentration of dissolved oxygen from the measured parameters.

Calibration of glassware

The pipet used to dispense iodate standards, the volumetric used to prepare the iodate standard, and oxygen s used in whole bottle titrations must be calibrated by weighing the weight of pure water dispensed or contained. The calibration must include a buoyancy correction.

None of the volumetric s used in the present intercomparison were calibrated.

Effect of temperature on mass of thiosulfate and iodate dispensed

Due to the thermal expansion of water, the mass, of the thiosulfate titrant and of the iodate standard dispensed by volumetric apparatus, depends on the solution temperature. Consequently, the temperature of the thiosulfate and iodate solutions at the time of delivery must be recorded, so that the solution concentrations can be reduced to a uniform temperature of 20°C.

Preparation of iodate standard

The standard should be prepared from reagent grade (primary standard grade if available) KIO_3 or $KH(IO_3)_2$. If possible, a common batch of KIO_3 should be distributed for use as a standard. Commercial iodate solutions must not be used as standards.

The weight of solids should be corrected for buoyancy, and 1987 atomic weights used to calculate the concentration (KIO_3 = 214.001; $KH(IO_3)_2$ = 389.912).

The volumetric concentration of the standard must be reduced to 20°C; this requires knowledge of the temperature at which the standard was prepared. Furthermore, the temperature at which the standard is dispensed must be measured so that the volume dispensed can be reduced to 20°C.

Standardization

Carpenter's (1965) recommended standardization procedure should be modified to include the addition of 1 ml of the $MnCl_2$ reagent after the 1 ml NaOH/NaI addition. The solution should be stirred between the NaOH/NaI and $MnCl_2$ additions.

Use of seawater blanks

Until more is known about the magnitude and variability of seawater blanks, only pure water blanks should be used in equations 1 and 3. Although this will cause small errors in the calculated oxygen concentration, it will insure that oxygens from different institutions are internally consistent.

Set of test calculations

Every institution performing WOCE oxygen measurements should be required to complete a set of test calculations, to check the accuracy of their data reduction techniques.

This set of test calculations should include: (1) data for calibration of a volumetric pipet; (2) data for calibration of an oxygen ; (3) data for calculation of the concentration of an iodate standard; and (4) sample titration data for low, medium, and high oxygen concentrations. Successful completion of the test calculations will insure that calculated results from all institutions are compatible.

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List of Figures

Appendix A. Dissolved oxygen concentrations measured by each institution during the dissolved oxygen intercalibration. Leg 3, R/V Oceanus cruise 219.

Cast	Niskin	Press	CTD	Salinity	Dissolved oxygen concentration (mL/L)							
					Bedford		Delaware		Scripps		Woods Hole	
#	#	(db)	Temp(C)	(PSU)	#1	#2	#1	#2	#1	#2	#1	#2
13	12	20	20.47	36.784	5.141	5.132	5.126	5.129	5.154	5.151	5.131	5.132
13	11	20	20.46	36.784	5.131	5.148	5.124	5.121	5.151	5.153	5.140	5.136
13	10	614	15.77	36.125	4.175	4.166	4.160	4.159	4.182	4.179	4.163	4.163
13	9	714	14.02	35.399	3.408	3.397	3.375	3.372	3.391	3.398	3.379	3.376
13	8	862	10.89	35.398	3.386	3.398	3.370	3.374	3.395	3.389	3.371	3.405
13	7	861	10.91	35.394	3.379	3.388	3.369	3.372	3.386	3.386	3.370	3.366
13	6	1258	5.46	35.065	5.172	5.202	5.188	5.180	5.208	5.208	5.195	5.188
13	5	1509	4.63	35.024	5.739	5.734	5.735	5.728	5.754	5.758	5.757	5.830
13	4	2014	3.93	35.002	6.015	6.027	6.023	6.005	6.041	6.034	6.027	6.020
13	3	3535	2.54	34.917	6.189	6.184	6.175	6.175	6.204	6.211	6.191	6.197
13	2	5195	2.22	34.875	5.912	5.912	5.896	5.897	5.933	5.931	5.914	5.930
15	12	896	9.41	35.227	3.386	3.383	3.358	3.358	3.373	3.367	3.352	3.354
15	11	896	9.41	35.227	3.368	3.370	3.363	3.364	---	3.375	3.358	3.354
15	10	896	9.41	35.229	3.381	3.372	3.355	3.356	3.363	3.367	3.355	3.358
15	9	896	9.41	35.228	3.367	3.375	3.357	3.352	3.366	3.363	3.351	3.349
15	8	896	9.41	35.229	3.373	3.369	3.352	3.351	3.380	3.369	3.346	3.354
15	7	896	9.41	35.229	3.384	3.374	3.370	3.362	3.377	3.374	3.358	3.356
15	6	896	9.41	35.232	3.391	3.402	3.385	3.372	3.389	3.389	3.376	3.370
15	5	896	9.41	35.230	3.426	3.394	3.377	3.377	3.392	3.392	3.370	3.374
15	4	896	9.41	35.230	3.408	3.400	3.384	3.383	3.390	3.395	3.374	3.375
15	3	896	9.41	35.229	3.394	3.401	3.380	3.374	3.386	3.389	3.372	3.371
16	12	3550	2.49	34.916	---	---	6.181	6.184	6.204	6.194	6.181	6.180
16	11	3550	2.49	34.917	6.175	6.186	6.175	6.175	6.217	6.209	6.186	6.183
16	10	3550	2.49	34.916	6.193	6.191	6.177	6.177	6.209	6.214	6.179	6.178
16	9	3550	2.49	34.916	6.161	6.175	6.180	6.179	6.208	6.205	6.181	6.188
16	8	3550	2.49	34.917	6.153	6.175	6.179	6.181	6.208	6.201	6.179	6.175
16	7	3549	2.49	34.915	6.175	6.188	6.183	6.179	6.211	6.210	6.182	6.181
16	6	3549	2.49	34.915	6.197	6.175	6.183	6.179	6.201	6.206	6.182	6.179
16	5	3550	2.49	34.915	6.180	6.187	6.180	6.183	6.211	6.214	6.178	6.180

Appendix A (continued).

Cast #	Niskin #	Press (db)	CTD Temp(C)	Salinity (PSU)	Dissolved oxygen concentration (mL/L)							
					Bedford		Delaware		Scripps		Woods Hole	
					#1	#2	#1	#2	#1	#2	#1	#2
16	4	3550	2.49	34.916	6.187	6.199	6.186	6.185	6.220	6.211	6.186	6.183
16	3	3549	2.49	34.916	6.211	6.176	6.177	6.179	6.212	6.207	6.168	6.185
20	12	23	20.19	36.720	5.197	5.203	5.202	5.207	5.227	5.223	5.203	5.206
20	11	22	20.19	36.719	5.208	5.199	5.198	5.193	5.232	5.227	5.212	5.211
20	10	213	18.74	36.598	4.869	4.865	4.864	4.862	4.891	4.887	4.867	4.870
20	9	415	17.78	36.463	4.500	4.503	4.508	4.502	4.528	4.525	4.508	4.505
20	8	616	16.00	36.170	4.204	4.205	4.200	4.199	4.215	4.211	4.194	4.196
20	7	939	8.92	35.192			3.524	3.521	3.532	3.534	3.518	3.514
20	6	2014	3.93	34.994			6.012	6.009	6.035	6.043	6.021	6.019
20	5	3029	2.95	34.946			6.109	6.108	6.139	6.132	6.110	6.110
20	4	4050	2.33	34.905			6.167	6.154	6.185	6.186	6.160	6.157
20	3	5109	2.26	34.887			5.990	5.988	6.017	6.006	5.981	5.982
21	12	3040	3.01	34.945			6.124	---	6.147	6.143	6.128	6.128
21	11	3247	2.80	34.926			6.161	6.156	6.186	6.177	6.175	6.182
21	10	3449	2.62	34.924			6.165	6.160	6.187	6.186	6.162	6.167
21	9	3654	2.47	34.915			6.179	6.174	6.134	6.198	6.177	6.181
21	8	3862	2.37	34.907			6.181	6.177	6.206	6.197	6.175	6.177
21	7	4067	2.30	34.900			6.147	---	6.171	6.170	6.153	6.153
21	6	4271	2.28	34.897			6.122	6.117	6.138	6.139	6.123	6.121
21	5	4472	2.26	34.893			6.099	6.100	6.120	6.120	6.103	6.102
21	4	4686	2.25	34.889			6.056	6.064	6.079	6.075	6.065	6.061
21	3	4945	2.24	34.883			5.986	5.986	6.010	6.003	5.988	5.986
23	12	11	19.51	36.632			5.358	---	5.377	5.371	5.364	5.365
23	11	107	18.80	36.621			5.031	5.036	5.059	5.048	5.035	5.039
23	10	204	18.68	36.609			5.003	5.006	5.024	5.024	5.000	5.008
23	9	310	18.13	36.512			4.559	4.556	4.563	4.569	4.552	4.554
23	8	407	17.66	36.446			4.510	---	4.528	4.526	4.513	4.516
23	7	512	16.85	36.315			4.292	4.288	4.302	4.304	4.283	4.288
23	6	624	15.56	36.095			4.173	4.176	4.182	4.187	4.171	4.172

Appendix A (continued).

Cast #	Niskin #	Press (db)	CTD Temp(C)	Salinity (PSU)	Dissolved oxygen concentration (mL/L)							
					Bedford		Delaware		Scripps		Woods Hole	
					#1	#2	#1	#2	#1	#2	#1	#2
23	5	715	14.00	35.841			3.892	3.893	3.897	3.901	3.884	---
23	4	812	12.18	35.564			3.445	3.444	3.457	3.444	3.436	3.439
23	3	909	10.06	35.305			3.383	3.425	3.381	3.376	3.368	3.374
24	12	1014	8.44	35.142			3.458	3.457	3.473	3.466	3.450	3.455
24	11	1216	5.64	35.046			4.967	4.967	4.986	4.983	4.952	4.977
24	10	1417	4.75	35.012			5.606	5.604	5.626	5.632	5.611	5.615
24	9	1622	4.34	34.991			5.879	5.873	5.898	5.900	5.884	5.882
24	8	1818	4.06	34.978			6.029	6.026	6.054	6.049	6.030	6.045
24	7	2025	3.89	34.980			6.064	6.061	6.089	6.079	6.065	6.065
24	6	2227	3.74	34.978			6.055	6.050	6.075	6.073	6.052	6.055
24	5	2429	3.57	34.977			6.043	6.045	6.066	6.063	6.046	6.044
24	4	2632	3.38	34.976			6.058	6.055	6.071	6.077	6.051	6.054
24	3	2840	3.19	34.959			6.082	6.077	6.110	6.102	6.080	6.080

Appendix B. Bedford Institute of Oceanography. Experimental data for the calculation of dissolved oxygen. Leg 3, R/V Oceanus cruise 219.

Iodate normality: 0.009952
Iodate pipet volume (mL): 9.96560

Thiosulfate titer (mL) for standards: 1.74900
Thiosulfate titer (mL) for blanks: 0.01275

Cast #	Niskin #	Pressure (db)	Oxygen (mL/L)	Thiosulfate Titer (mL)	--- Flask --- #	Volume (mL)
13	12	20	5.141	2.1052	728	131.82
13	12	20	5.132	2.1013	773	131.80
13	11	20	5.131	2.0994	737	131.71
13	11	20	5.148	2.1269	781	133.00
13	10	614	4.175	1.6813	700	129.40
13	10	614	4.166	1.7050	740	131.47
13	9	714	3.408	1.3984	736	131.50
13	9	714	3.397	1.3567	382	128.02
13	8	862	3.386	1.3454	524	127.35
13	8	862	3.398	1.3994	705	132.00
13	7	861	3.379	1.3709	835	130.01
13	7	861	3.388	1.3980	844	132.26
13	6	1258	5.172	2.1416	362	133.29
13	6	1258	5.202	2.1159	767	130.95
13	5	1509	5.739	2.3510	704	131.99
13	5	1509	5.734	2.2976	761	129.11
13	4	2014	6.015	2.4646	756	132.07
13	4	2014	6.027	2.4247	758	129.68
13	3	3535	6.189	2.4748	817	128.93
13	3	3535	6.184	2.5056	608	130.62
13	2	5195	5.912	2.4414	678	133.07
13	2	5195	5.912	2.3820	853	129.86
15	12	896	3.386	1.4084	362	133.29
15	12	896	3.383	1.3821	767	130.95
15	11	896	3.368	1.3629	758	129.68
15	11	896	3.370	1.3888	756	132.07
15	10	896	3.381	1.3779	608	130.62
15	10	896	3.372	1.3565	817	128.93
15	9	896	3.367	1.3980	678	133.07
15	9	896	3.375	1.3675	853	129.86
15	8	896	3.373	1.3403	524	127.35
15	8	896	3.369	1.3875	705	132.00
15	7	896	3.384	1.3919	728	131.82
15	7	896	3.374	1.3877	773	131.80
15	6	896	3.391	1.4071	781	133.00
15	6	896	3.402	1.3979	737	131.71
15	5	896	3.426	1.4050	740	131.47
15	5	896	3.394	1.3705	700	129.40
15	4	896	3.408	1.3980	736	131.50
15	4	896	3.400	1.3579	382	128.02
15	3	896	3.394	1.3817	848	130.48
15	3	896	3.401	1.4071	709	132.59

Appendix B (continued).

Cast #	Niskin #	Pressure (db)	Oxygen (mL/L)	Thiosulfate Titer (mL)	--- Flask --- #	Volume (mL)
16	11	3550	6.175	2.5476	781	133.00
16	11	3550	6.186	2.5274	737	131.71
16	10	3550	6.193	2.5470	709	132.59
16	10	3550	6.191	2.5055	848	130.48
16	9	3550	6.161	2.5125	740	131.47
16	9	3550	6.175	2.4784	700	129.40
16	8	3550	6.153	2.5195	704	131.99
16	8	3550	6.175	2.4727	761	129.11
16	7	3549	6.175	2.5534	362	133.29
16	7	3549	6.188	2.5135	767	130.95
16	6	3549	6.197	2.5387	756	132.07
16	6	3549	6.175	2.4837	758	129.68
16	5	3550	6.180	2.5354	844	132.26
16	5	3550	6.187	2.4950	835	130.01
16	4	3550	6.187	2.4741	817	128.93
16	4	3550	6.199	2.5114	608	130.62
16	3	3549	6.211	2.5639	678	133.07
16	3	3549	6.176	2.4876	853	129.86
20	12	23	5.197	2.1318	756	132.07
20	12	23	5.203	2.0956	758	129.68
20	11	22	5.208	2.1350	704	131.99
20	11	22	5.199	2.0848	761	129.11
20	10	213	4.869	1.9507	817	128.93
20	10	213	4.865	1.9634	853	129.86
20	9	415	4.500	1.8281	608	130.62
20	9	415	4.503	1.8640	678	133.07
20	8	616	4.204	1.6930	700	129.40
20	8	616	4.205	1.7207	740	131.47

Appendix C. University of Delaware. Experimental data for the calculation of dissolved oxygen. Leg 3, R/V Oceanus cruise 219.

Iodate normality: 0.010140
Iodate pipet volume (mL): 9.97840

Thiosulfate titer (mL) for standards: 0.71800
Thiosulfate titer (mL) for blanks: -0.00040

Cast #	Niskin #	Pressure (db)	Oxygen (mL/L)	Thiosulfate Titer (mL)	-- Flask -- #	Volume (mL)
13	12	20	5.126	0.9232	35	143.73
13	12	20	5.129	0.9468	36	147.26
13	11	20	5.124	0.9581	41	149.16
13	11	20	5.121	0.9431	43	146.92
13	10	614	4.160	0.7733	39	148.23
13	10	614	4.159	0.7633	40	146.38
13	9	714	3.375	0.6265	37	147.93
13	9	714	3.372	0.6228	38	147.22
13	8	862	3.370	0.5850	17	138.47
13	8	862	3.374	0.6202	19	146.53
13	7	861	3.369	0.5880	30	139.20
13	7	861	3.372	0.6188	34	146.26
13	6	1258	5.188	0.9059	23	139.41
13	6	1258	5.180	0.9540	24	146.93
13	5	1509	5.735	1.0031	20	139.66
13	5	1509	5.728	1.0538	21	146.81
13	4	2014	6.023	1.1123	1	147.38
13	4	2014	6.005	1.1068	2	147.09
13	3	3535	6.175	1.0892	11	140.85
13	3	3535	6.175	1.1197	13	144.74
13	2	5195	5.896	1.0261	4	138.97
13	2	5195	5.897	1.0886	7	147.30
15	12	896	3.358	0.5861	30	139.20
15	12	896	3.358	0.6161	34	146.26
15	11	896	3.363	0.6255	39	148.23
15	11	896	3.364	0.6178	40	146.38
15	10	896	3.355	0.6228	37	147.93
15	10	896	3.356	0.6199	38	147.22
15	9	896	3.357	0.6052	35	143.73
15	9	896	3.352	0.6194	36	147.26
15	8	896	3.352	0.5921	11	140.85
15	8	896	3.351	0.6085	13	144.74
15	7	896	3.370	0.5890	23	139.41
15	7	896	3.362	0.6198	24	146.93
15	6	896	3.385	0.5927	20	139.66
15	6	896	3.372	0.6211	21	146.81
15	5	896	3.377	0.5862	17	138.47
15	5	896	3.377	0.6208	19	146.53
15	4	896	3.384	0.6257	1	147.38
15	4	896	3.383	0.6243	2	147.09
15	3	896	3.380	0.5890	4	138.97
15	3	896	3.374	0.6236	7	147.30

Appendix C (continued).

Cast #	Niskin #	Pressure (db)	Oxygen (mL/L)	Thiosulfate Titer (mL)	-- Flask -- #	Volume (mL)
16	12	3550	6.181	1.0773	30	139.20
16	12	3550	6.184	1.1333	34	146.26
16	11	3550	6.175	1.1470	39	148.23
16	11	3550	6.175	1.1325	40	146.38
16	10	3550	6.177	1.1450	37	147.93
16	10	3550	6.177	1.1395	38	147.22
16	9	3550	6.180	1.1126	35	143.73
16	9	3550	6.179	1.1401	36	147.26
16	8	3550	6.179	1.0900	11	140.85
16	8	3550	6.181	1.1207	13	144.74
16	7	3549	6.183	1.0793	23	139.41
16	7	3549	6.179	1.1376	24	146.93
16	6	3549	6.183	1.0813	20	139.66
16	6	3549	6.179	1.1366	21	146.81
16	5	3550	6.180	1.0715	17	138.47
16	5	3550	6.183	1.1352	19	146.53
16	4	3550	6.186	1.1424	1	147.38
16	4	3550	6.185	1.1400	2	147.09
16	3	3549	6.177	1.0749	4	138.97
16	3	3549	6.179	1.1405	7	147.30
20	12	23	5.202	0.9070	30	139.20
20	12	23	5.207	0.9545	34	146.26
20	11	22	5.198	0.9658	39	148.23
20	11	22	5.193	0.9528	40	146.38
20	10	213	4.864	0.9020	37	147.93
20	10	213	4.862	0.8973	38	147.22
20	9	415	4.508	0.8122	35	143.73
20	9	415	4.502	0.8312	36	147.26
20	8	616	4.200	0.7414	11	140.85
20	8	616	4.199	0.7619	13	144.74
20	7	939	3.524	0.6159	23	139.41
20	7	939	3.521	0.6490	24	146.93
20	6	2014	6.012	1.0514	20	139.66
20	6	2014	6.009	1.1055	21	146.81
20	5	3029	6.109	1.0592	17	138.47
20	5	3029	6.108	1.1215	19	146.53
20	4	4050	6.167	1.1389	1	147.38
20	4	4050	6.154	1.1343	2	147.09
20	3	5109	5.990	1.0423	4	138.97
20	3	5109	5.988	1.1052	7	147.30
21	12	3040	6.124	1.1223	34	146.26
21	11	3247	6.161	1.1445	39	148.23
21	11	3247	6.156	1.1290	40	146.38
21	10	3449	6.165	1.1429	37	147.93
21	10	3449	6.160	1.1364	38	147.22
21	9	3654	6.179	1.1125	35	143.73
21	9	3654	6.174	1.1393	36	147.26
21	8	3862	6.181	1.0903	11	140.85
21	8	3862	6.177	1.1201	13	144.74

Appendix C (continued).

Cast #	Niskin #	Pressure (db)	Oxygen (mL/L)	Thiosulfate Titer (mL)	-- Flask -- #	Volume (mL)
21	7	4067	6.147	1.1317	24	146.93
21	6	4271	6.122	1.0707	20	139.66
21	6	4271	6.117	1.1252	21	146.81
21	5	4472	6.099	1.0574	17	138.47
21	5	4472	6.100	1.1200	19	146.53
21	4	4686	6.056	1.1184	1	147.38
21	4	4686	6.064	1.1177	2	147.09
21	3	4945	5.986	1.0416	4	138.97
21	3	4945	5.986	1.1049	7	147.30
23	12	11	5.358	0.9822	34	146.26
23	11	107	5.031	0.9348	39	148.23
23	11	107	5.036	0.9239	40	146.38
23	10	204	5.003	0.9278	37	147.93
23	10	204	5.006	0.9238	38	147.22
23	9	310	4.559	0.8213	35	143.73
23	9	310	4.556	0.8412	36	147.26
23	8	407	4.510	0.8308	24	146.93
23	7	512	4.292	0.7512	20	139.66
23	7	512	4.288	0.7894	21	146.81
23	6	624	4.173	0.7241	17	138.47
23	6	624	4.176	0.7673	19	146.53
23	5	715	3.892	0.6872	11	140.85
23	5	715	3.893	0.7065	13	144.74
23	4	812	3.445	0.6370	1	147.38
23	4	812	3.444	0.6356	2	147.09
23	3	909	3.383	0.5894	4	138.97
23	3	909	3.425	0.6329	7	147.30
24	12	1014	3.458	0.6035	30	139.20
24	12	1014	3.457	0.6342	34	146.26
24	11	1216	4.967	0.9288	41	149.16
24	11	1216	4.967	0.9147	43	146.92
24	10	1417	5.606	1.0394	37	147.93
24	10	1417	5.604	1.0340	38	147.22
24	9	1622	5.879	1.0586	35	143.73
24	9	1622	5.873	1.0838	36	147.26
24	8	1818	6.029	1.0635	11	140.85
24	8	1818	6.026	1.0927	13	144.74
24	7	2025	6.064	1.0586	23	139.41
24	7	2025	6.061	1.1158	24	146.93
24	6	2227	6.055	1.0589	20	139.66
24	6	2227	6.050	1.1129	21	146.81
24	5	2429	6.043	1.0477	17	138.47
24	5	2429	6.045	1.1099	19	146.53
24	4	2632	6.058	1.1188	1	147.38
24	4	2632	6.055	1.1160	2	147.09
24	3	2840	6.082	1.0584	4	138.97
24	3	2840	6.077	1.1217	7	147.30

Appendix D. Scripps Institution of Oceanography. Experimental data for the calculation of dissolved oxygen. Leg 3, R/V Oceanus cruise 219.

Iodate normality: 0.010000
Iodate pipet volume (mL): 10.00000

Thiosulfate titer (mL) for standards: 0.70935
Thiosulfate titer (mL) for blanks: -0.00065

Cast #	Niskin #	Pressure (db)	Oxygen (mL/L)	Thiosulfate Titer (mL)	--- Flask --- #	Volume (mL)
13	12	20	5.154	0.6612	560	102.92
13	12	20	5.151	0.6503	577	101.31
13	11	20	5.151	0.6738	557	104.90
13	11	20	5.153	0.6586	576	102.54
13	10	614	4.182	0.5287	556	101.40
13	10	614	4.179	0.5362	573	102.88
13	9	714	3.391	0.4438	555	104.83
13	9	714	3.398	0.4403	572	103.82
13	8	862	3.395	0.4251	554	100.37
13	8	862	3.389	0.4602	571	108.71
13	7	861	3.386	0.4426	552	104.70
13	7	861	3.386	0.4150	569	98.29
13	6	1258	5.208	0.6889	551	106.07
13	6	1258	5.208	0.6519	568	100.47
13	5	1509	5.754	0.7517	514	104.79
13	5	1509	5.758	0.7327	567	102.13
13	4	2014	6.041	0.8224	513	109.14
13	4	2014	6.034	0.7578	565	100.82
13	3	3535	6.204	0.7836	490	101.40
13	3	3535	6.211	0.7804	564	100.87
13	2	5195	5.933	0.7243	418	98.05
13	2	5195	5.931	0.7503	563	101.54
15	12	896	3.373	0.4332	560	102.92
15	12	896	3.367	0.4256	577	101.31
15	11	896	3.375	0.4319	576	102.54
15	10	896	3.363	0.4255	556	101.40
15	10	896	3.367	0.4323	573	102.88
15	9	896	3.366	0.4405	555	104.83
15	9	896	3.363	0.4358	572	103.82
15	8	896	3.380	0.4232	554	100.37
15	8	896	3.369	0.4575	571	108.71
15	7	896	3.377	0.4414	552	104.70
15	7	896	3.374	0.4135	569	98.29
15	6	896	3.389	0.4489	551	106.07
15	6	896	3.389	0.4248	568	100.47
15	5	896	3.392	0.4437	514	104.79
15	5	896	3.392	0.4322	567	102.13
15	4	896	3.390	0.4621	513	109.14
15	4	896	3.395	0.4270	565	100.82
15	3	896	3.386	0.4284	490	101.40
15	3	896	3.389	0.4265	564	100.87

Appendix D (continued).

Cast #	Niskin #	Pressure (db)	Oxygen (mL/L)	Thiosulfate Titer (mL)	--- Flask --- #	Volume (mL)
16	12	3550	6.204	0.8785	273	113.45
16	12	3550	6.194	0.7669	15	99.42
16	11	3550	6.217	0.7794	207	100.66
16	11	3550	6.209	0.8472	340	109.39
16	10	3550	6.209	0.7603	176	98.35
16	10	3550	6.214	0.7743	333	100.06
16	9	3550	6.208	0.8307	166	107.31
16	9	3550	6.205	0.8723	332	112.65
16	8	3550	6.208	0.7908	152	102.25
16	8	3550	6.201	0.7828	331	101.34
16	7	3549	6.211	0.8558	150	110.45
16	7	3549	6.210	0.7575	329	97.99
16	6	3549	6.201	0.8489	143	109.74
16	6	3549	6.206	0.7596	327	98.32
16	5	3550	6.211	0.7899	87	102.09
16	5	3550	6.214	0.7975	315	102.99
16	4	3550	6.220	0.7730	44	99.80
16	4	3550	6.211	0.7943	303	102.65
16	3	3549	6.212	0.8671	32	111.87
16	3	3549	6.207	0.8451	297	109.16
20	12	23	5.227	0.6706	560	102.92
20	12	23	5.223	0.6594	577	101.31
20	11	22	5.232	0.6844	557	104.90
20	11	22	5.227	0.6680	576	102.54
20	10	213	4.891	0.6181	556	101.40
20	10	213	4.887	0.6268	573	102.88
20	9	415	4.528	0.5921	555	104.83
20	9	415	4.525	0.5859	572	103.82
20	8	616	4.215	0.5274	554	100.37
20	8	616	4.211	0.5714	571	108.71
20	7	939	3.532	0.4616	552	104.70
20	7	939	3.534	0.4331	569	98.29
20	6	2014	6.035	0.7981	551	106.07
20	6	2014	6.043	0.7562	568	100.47
20	5	3029	6.139	0.8018	514	104.79
20	5	3029	6.132	0.7802	567	102.13
20	4	4050	6.185	0.8419	513	109.14
20	4	4050	6.186	0.7768	565	100.82
20	3	5109	6.017	0.7601	490	101.40
20	3	5109	6.006	0.7547	564	100.87
21	12	3040	6.147	0.8704	273	113.45
21	12	3040	6.143	0.7606	15	99.42
21	11	3247	6.186	0.7756	207	100.66
21	11	3247	6.177	0.8428	340	109.39
21	10	3449	6.187	0.7576	176	98.35
21	10	3449	6.186	0.7708	333	100.06
21	9	3654	6.134	0.8207	166	107.31
21	9	3654	6.198	0.8713	332	112.65
21	8	3862	6.206	0.7906	152	102.25
21	8	3862	6.197	0.7823	331	101.34

Appendix D (continued).

Cast #	Niskin #	Pressure (db)	Oxygen (mL/L)	Thiosulfate Titer (mL)	--- Flask --- #	Volume (mL)
21	7	4067	6.171	0.8503	150	110.45
21	7	4067	6.170	0.7526	329	97.99
21	6	4271	6.138	0.8402	143	109.74
21	6	4271	6.139	0.7514	327	98.32
21	5	4472	6.120	0.7784	87	102.09
21	5	4472	6.120	0.7854	315	102.99
21	4	4686	6.079	0.7555	44	99.80
21	4	4686	6.075	0.7770	303	102.65
21	3	4945	6.010	0.8390	32	111.87
21	3	4945	6.003	0.8174	297	109.16
23	12	11	5.377	0.7616	273	113.45
23	12	11	5.371	0.6652	15	99.42
23	11	107	5.059	0.6346	207	100.66
23	11	107	5.048	0.6890	340	109.39
23	10	204	5.024	0.6154	176	98.35
23	10	204	5.024	0.6263	333	100.06
23	9	310	4.563	0.6110	166	107.31
23	9	310	4.569	0.6428	332	112.65
23	8	407	4.528	0.5772	152	102.25
23	8	407	4.526	0.5718	331	101.34
23	7	512	4.302	0.5932	150	110.45
23	7	512	4.304	0.5255	329	97.99
23	6	624	4.182	0.5730	143	109.74
23	6	624	4.187	0.5130	327	98.32
23	5	715	3.897	0.4962	87	102.09
23	5	715	3.901	0.5012	315	102.99
23	4	812	3.457	0.4303	44	99.80
23	4	812	3.444	0.4412	303	102.65
23	3	909	3.381	0.4727	32	111.87
23	3	909	3.376	0.4604	297	109.16
24	12	1014	3.473	0.4461	560	102.92
24	12	1014	3.466	0.4381	577	101.31
24	11	1216	4.986	0.6523	557	104.90
24	11	1216	4.983	0.6369	576	102.54
24	10	1417	5.626	0.7108	556	101.40
24	10	1417	5.632	0.7221	573	102.88
24	9	1622	5.898	0.7707	555	104.83
24	9	1622	5.900	0.7634	572	103.82
24	8	1818	6.054	0.7568	554	100.37
24	8	1818	6.049	0.8202	571	108.71
24	7	2025	6.089	0.7946	552	104.70
24	7	2025	6.079	0.7439	569	98.29
24	6	2227	6.075	0.8034	551	106.07
24	6	2227	6.073	0.7600	568	100.47
24	5	2429	6.066	0.7924	514	104.79
24	5	2429	6.063	0.7715	567	102.13
24	4	2632	6.071	0.8264	513	109.14
24	4	2632	6.077	0.7632	565	100.82
24	3	2840	6.110	0.7718	490	101.40
24	3	2840	6.102	0.7667	564	100.87

Appendix E. Woods Hole Oceanographic Institution. Experimental data for the calculation of dissolved oxygen. Leg 3, R/V Oceanus cruise 219.

winkler titration values

Cruise id..... OC 219
Station #..... 13
Case id..... J
Stdze. thio..... 4.408
Normality..... 1.009585E-02
Volume of automatic burette..... 49.858
Volume of biniodate used to standardize... 15
Average volume of sample bottles..... 147.5
Normality of biniodate solution..... .01
Volume of distilled water dispensor..... 150.153
Operator's name..... GPK
Date & Time..... 06-15-1990 11:50:26

#	Begin volt	End volt	thio	oxygen
1	19.98	0.02	5.123	5.872
2	20.23	0.02	5.160	5.914
3	22.64	0.02	5.401	6.191
4	21.23	0.02	5.258	6.027
5	21.38	0.02	5.023	5.757
6	18.79	0.02	4.534	5.195
7	18.13	0.02	2.946	3.370
8	18.23	0.02	2.947	3.371
9	16.74	0.02	2.954	3.379
10	14.49	0.02	3.636	4.163
11	18.84	0.02	4.486	5.140
12	18.87	0.02	4.478	5.131
13	20.62	0.02	0.000	-9.000
14	21.54	0.02	5.174	5.930
15	0.00	0.00	5.406	6.197
16	21.34	0.02	5.252	6.020
17	19.93	0.02	5.087	5.830
18	18.58	0.02	4.528	5.188
19	14.12	0.02	2.942	3.366
20	18.07	0.02	2.976	3.405
21	18.92	0.02	2.951	3.376
22	19.53	0.02	3.636	4.163
23	19.57	0.02	4.483	5.136
24	19.68	0.02	4.479	5.132
25	0.00	0.00	0.000	-9.000
26	0.00	0.00	0.000	-9.000
27	0.00	0.00	0.000	-9.000
28	0.00	0.00	0.000	-9.000
29	0.00	0.00	0.000	-9.000
30	0.00	0.00	0.000	-9.000
31	0.00	0.00	0.000	-9.000
32	0.00	0.00	0.000	-9.000
33	0.00	0.00	0.000	-9.000
34	0.00	0.00	0.000	-9.000
35	0.00	0.00	0.000	-9.000
36	0.00	0.00	0.000	-9.000

winkler titration values

Cruise id..... OC 219
Station #..... 15
Case id..... J
Stdze. thio..... 4.414
Normality..... 1.008212E-02
Volume of automatic burette..... 49.858
Volume of biniodate used to standardize... 15
Average volume of sample bottles..... 147.5
Normality of biniodate solution..... .01
Volume of distilled water dispensor..... 150.153
Operator's name..... GPK
Date & Time..... 06-15-1990 11:50:03

#	Begin volt	End volt	thio	oxygen
1	18.48	0.02	3.009	3.438
2	17.53	0.02	2.947	3.367
3	18.05	0.02	2.952	3.372
4	16.09	0.02	2.953	3.374
5	18.56	0.02	2.950	3.370
6	16.96	0.02	2.955	3.376
7	15.65	0.02	2.939	3.358
8	19.86	0.02	2.929	3.346
9	20.22	0.02	2.933	3.351
10	15.19	0.02	2.937	3.355
11	17.91	0.02	2.939	3.358
12	18.11	0.02	2.934	3.352
13	15.86	0.02	0.000	-9.000
14	0.00	0.00	0.000	-9.000
15	0.00	0.00	2.951	3.371
16	14.29	0.02	2.954	3.375
17	18.65	0.02	2.953	3.374
18	17.46	0.02	2.950	3.370
19	18.02	0.02	2.938	3.356
20	15.97	0.02	2.936	3.354
21	16.58	0.02	2.932	3.349
22	16.42	0.02	2.939	3.358
23	17.62	0.02	2.936	3.354
24	17.99	0.02	2.936	3.354
25	0.00	0.00	0.000	-9.000
26	0.00	0.00	0.000	-9.000
27	0.00	0.00	0.000	-9.000
28	0.00	0.00	0.000	-9.000
29	0.00	0.00	0.000	-9.000
30	0.00	0.00	0.000	-9.000
31	0.00	0.00	0.000	-9.000
32	0.00	0.00	0.000	-9.000
33	0.00	0.00	0.000	-9.000
34	0.00	0.00	0.000	-9.000
35	0.00	0.00	0.000	-9.000
36	0.00	0.00	0.000	-9.000

winkler titration values

Cruise id..... OC 219
Station #..... 16
Case id..... S
Stdze. thio..... 4.414
Normality..... 1.008212E-02
Volume of automatic burette..... 49.858
Volume of biniodate used to standardize... 15
Average volume of sample bottles..... 148.8
Normality of biniodate solution..... .01
Volume of distilled water dispensor..... 150.153
Operator's name..... GPK
Date & Time..... 06-15-1990 11:49:42

#	Begin volt	End volt	thio	oxygen
1	0.00	0.00	0.000	-9.000
2	0.00	0.00	0.000	-9.000
3	19.59	0.02	5.389	6.168
4	22.60	0.02	5.404	6.186
5	19.32	0.02	5.397	6.178
6	15.84	0.02	5.401	6.182
7	23.40	0.02	5.401	6.182
8	22.73	0.02	5.398	6.179
9	21.16	0.02	5.400	6.181
10	21.58	0.02	5.398	6.179
11	20.03	0.02	5.404	6.186
12	22.34	0.02	5.400	6.181
13	0.00	0.00	0.000	-9.000
14	0.00	0.00	0.000	-9.000
15	21.14	0.02	5.403	6.185
16	20.23	0.02	5.402	6.183
17	21.26	0.02	5.399	6.180
18	20.01	0.02	5.398	6.179
19	22.09	0.02	5.400	6.181
20	23.24	0.02	5.395	6.175
21	22.57	0.02	5.406	6.188
22	21.34	0.02	5.397	6.178
23	22.30	0.02	5.402	6.183
24	22.17	0.02	5.399	6.180
25	0.00	0.00	0.000	-9.000
26	0.00	0.00	0.000	-9.000
27	0.00	0.00	0.000	-9.000
28	0.00	0.00	0.000	-9.000
29	0.00	0.00	0.000	-9.000
30	0.00	0.00	0.000	-9.000
31	0.00	0.00	0.000	-9.000
32	0.00	0.00	0.000	-9.000
33	0.00	0.00	0.000	-9.000
34	0.00	0.00	0.000	-9.000
35	0.00	0.00	0.000	-9.000
36	0.00	0.00	0.000	-9.000

winkler titration values

Cruise id..... OC 219
Station #..... 20
Case id..... J
Stdze. thio..... 4.413
Normality..... .0100844
Volume of automatic burette..... 49.858
Volume of biniodate used to standardize... 15
Average volume of sample bottles..... 147.5
Normality of biniodate solution..... .01
Volume of distilled water dispensor..... 150.153
Operator's name..... GPK
Date & Time..... 06-15-1990 11:48:47

#	Begin volt	End volt	thio	oxygen
1	0.00	0.00	0.000	-9.000
2	0.00	0.00	0.000	-9.000
3	22.80	0.02	5.224	5.981
4	23.53	0.02	5.380	6.160
5	22.83	0.02	5.336	6.110
6	21.14	0.02	5.259	6.021
7	19.20	0.02	3.078	3.518
8	21.21	0.02	3.667	4.194
9	19.29	0.02	3.941	4.508
10	22.63	0.02	4.253	4.867
11	22.52	0.02	4.554	5.212
12	22.68	0.02	4.546	5.203
13	0.00	0.00	0.000	-9.000
14	0.00	0.00	0.000	-9.000
15	23.68	0.02	5.225	5.982
16	23.50	0.02	5.377	6.157
17	22.77	0.02	5.336	6.110
18	23.17	0.02	5.257	6.019
19	20.42	0.02	3.075	3.514
20	21.93	0.02	3.669	4.196
21	21.64	0.02	3.938	4.505
22	16.13	0.02	4.256	4.870
23	20.32	0.02	4.553	5.211
24	19.27	0.02	4.549	5.206
25	0.00	0.00	0.000	-9.000
26	0.00	0.00	0.000	-9.000
27	0.00	0.00	0.000	-9.000
28	0.00	0.00	0.000	-9.000
29	0.00	0.00	0.000	-9.000
30	0.00	0.00	0.000	-9.000
31	0.00	0.00	0.000	-9.000
32	0.00	0.00	0.000	-9.000
33	0.00	0.00	0.000	-9.000
34	0.00	0.00	0.000	-9.000
35	0.00	0.00	0.000	-9.000
36	0.00	0.00	0.000	-9.000

winkler titration values

Cruise id..... OC 219
Station #..... 21
Case id..... J
Stdze. thio..... 4.413
Normality..... .0100844
Volume of automatic burette..... 49.858
Volume of biniodate used to standardize... 15
Average volume of sample bottles..... 147.5
Normality of biniodate solution..... .01
Volume of distilled water dispensor..... 150.153
Operator's name..... GPK
Date & Time..... 06-15-1990 11:48:20

#	Begin volt	End volt	thio	oxygen
1	20.48	0.02	5.195	5.948
2	21.75	0.02	5.228	5.986
3	20.10	0.02	5.230	5.988
4	19.89	0.02	5.297	6.065
5	20.52	0.02	5.330	6.103
6	22.09	0.02	5.348	6.123
7	22.58	0.02	5.374	6.153
8	23.76	0.02	5.393	6.175
9	17.06	0.02	5.395	6.177
10	20.05	0.02	5.382	6.162
11	19.43	0.02	5.393	6.175
12	22.42	0.02	5.352	6.128
13	0.00	0.00	0.000	-9.000
14	0.00	0.00	0.000	-9.000
15	20.83	0.02	5.228	5.986
16	21.00	0.02	5.294	6.061
17	20.92	0.03	5.329	6.102
18	23.24	0.02	5.346	6.121
19	17.43	0.02	5.374	6.153
20	20.35	0.02	5.395	6.177
21	17.35	0.02	5.398	6.181
22	20.65	0.02	5.386	6.167
23	21.64	0.02	5.399	6.182
24	20.60	0.02	5.352	6.128
25	0.00	0.00	0.000	-9.000
26	0.00	0.00	0.000	-9.000
27	0.00	0.00	0.000	-9.000
28	0.00	0.00	0.000	-9.000
29	0.00	0.00	0.000	-9.000
30	0.00	0.00	0.000	-9.000
31	0.00	0.00	0.000	-9.000
32	0.00	0.00	0.000	-9.000
33	0.00	0.00	0.000	-9.000
34	0.00	0.00	0.000	-9.000
35	0.00	0.00	0.000	-9.000
36	0.00	0.00	0.000	-9.000

winkler titration values

Cruise id..... OC 219
Station #..... 23
Case id..... S
Stdze. thio..... 4.414
Normality..... 1.008212E-02
Volume of automatic burette..... 49.858
Volume of biniodate used to standardize... 15
Average volume of sample bottles..... 148.8
Normality of biniodate solution..... .01
Volume of distilled water dispensor..... 150.153
Operator's name..... GPK
Date & Time..... 06-15-1990 11:47:32

#	Begin volt	End volt	thio	oxygen
1	17.17	0.02	2.993	3.419
2	17.25	0.02	2.956	3.377
3	17.71	0.02	2.948	3.368
4	16.64	0.02	3.008	3.436
5	17.10	0.02	3.398	3.884
6	17.54	0.02	3.648	4.171
7	14.90	0.02	3.746	4.283
8	16.51	0.02	3.946	4.513
9	20.48	0.02	3.980	4.552
10	22.94	0.02	4.371	5.000
11	19.96	0.02	4.401	5.035
12	21.30	0.02	4.688	5.364
13	0.00	0.00	0.000	-9.000
14	0.00	0.00	0.000	-9.000
15	16.91	0.02	2.954	3.374
16	16.96	0.02	3.010	3.439
17	0.00	0.00	0.000	-9.000
18	18.77	0.02	3.649	4.172
19	17.86	0.02	3.750	4.288
20	20.63	0.02	3.949	4.516
21	19.37	0.02	3.982	4.554
22	20.28	0.02	4.378	5.008
23	18.15	0.02	4.405	5.039
24	19.41	0.02	4.689	5.365
25	0.00	0.00	0.000	-9.000
26	0.00	0.00	0.000	-9.000
27	0.00	0.00	0.000	-9.000
28	0.00	0.00	0.000	-9.000
29	0.00	0.00	0.000	-9.000
30	0.00	0.00	0.000	-9.000
31	0.00	0.00	0.000	-9.000
32	0.00	0.00	0.000	-9.000
33	0.00	0.00	0.000	-9.000
34	0.00	0.00	0.000	-9.000
35	0.00	0.00	0.000	-9.000
36	0.00	0.00	0.000	-9.000

winkler titration values

Cruise id..... OC 219
Station #..... 24
Case id..... J
Stdze. thio..... 4.414
Normality..... 1.008212E-02
Volume of automatic burette..... 49.858
Volume of biniodate used to standardize... 15
Average volume of sample bottles..... 147.5
Normality of biniodate solution..... .01
Volume of distilled water dispensor..... 150.153
Operator's name..... GPK
Date & Time..... 06-15-1990 11:45:54

#	Begin volt	End volt	thio	oxygen
1	23.19	0.02	4.732	5.415
2	22.09	0.02	4.708	5.388
3	18.90	0.02	5.311	6.080
4	21.60	0.02	5.286	6.051
5	16.24	0.02	5.282	6.046
6	23.58	0.02	5.287	6.052
7	19.26	0.02	5.298	6.065
8	18.86	0.02	5.268	6.030
9	20.26	0.02	5.141	5.884
10	22.29	0.02	4.903	5.611
11	22.33	0.02	4.328	4.952
12	18.50	0.02	3.020	3.450
13	0.00	0.00	0.000	-9.000
14	0.00	0.00	0.000	-9.000
15	23.47	0.02	5.311	6.080
16	20.57	0.02	5.289	6.054
17	17.23	0.02	5.280	6.044
18	19.92	0.02	5.290	6.055
19	18.44	0.02	5.298	6.065
20	21.16	0.02	5.281	6.045
21	18.50	0.02	5.139	5.882
22	18.70	0.02	4.906	5.615
23	16.86	0.02	4.350	4.977
24	16.48	0.02	3.024	3.455
25	0.00	0.00	0.000	-9.000
26	0.00	0.00	0.000	-9.000
27	0.00	0.00	0.000	-9.000
28	0.00	0.00	0.000	-9.000
29	0.00	0.00	0.000	-9.000
30	0.00	0.00	0.000	-9.000
31	0.00	0.00	0.000	-9.000
32	0.00	0.00	0.000	-9.000
33	0.00	0.00	0.000	-9.000
34	0.00	0.00	0.000	-9.000
35	0.00	0.00	0.000	-9.000
36	0.00	0.00	0.000	-9.000

Figure 1. Stations occupied during the oxygen intercalibration. R/V Oceanus cruise 219, leg 3.

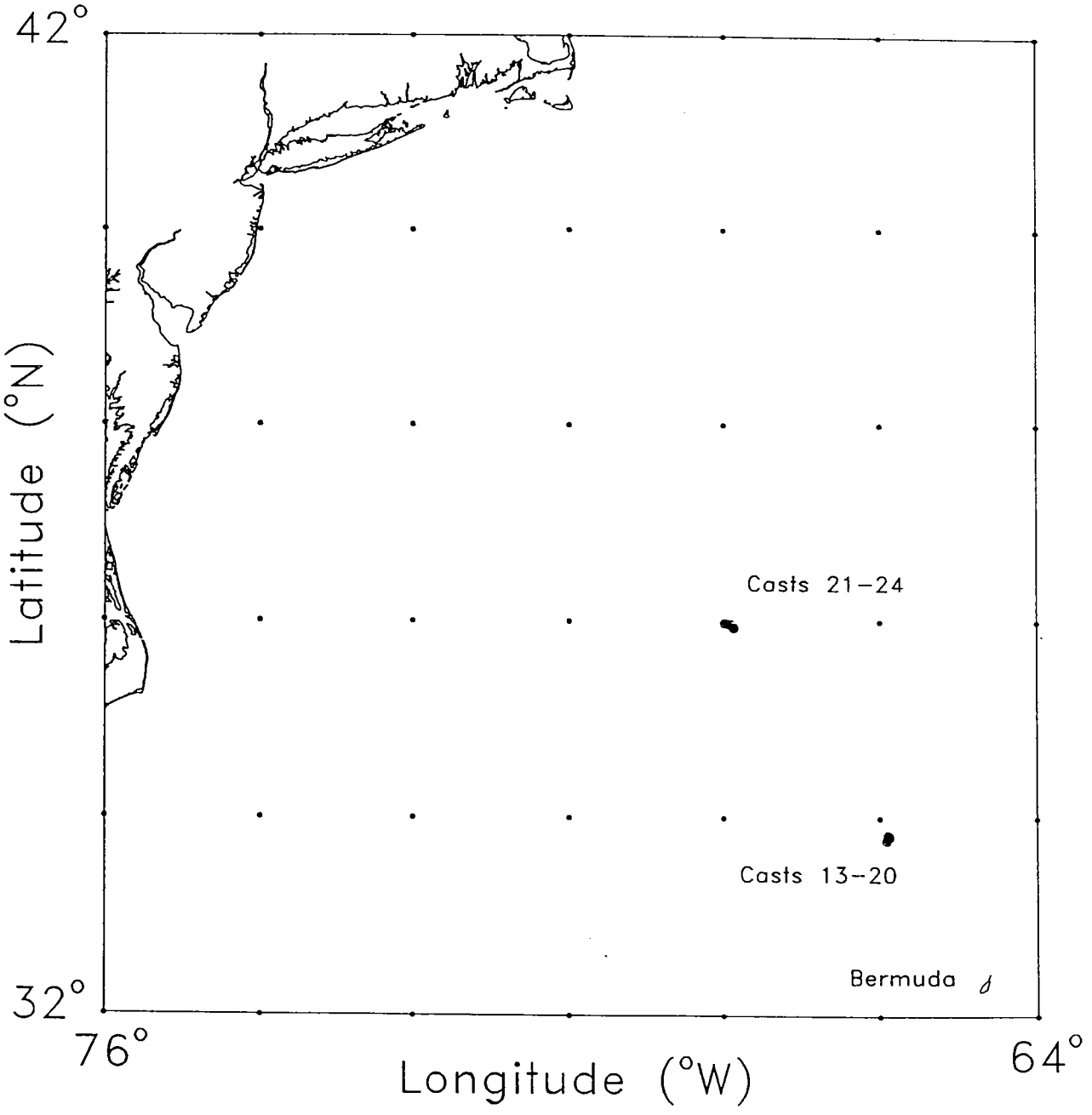

Figure 2. Hydrographic data at Station 1; casts 13, 15, 16, and 20.

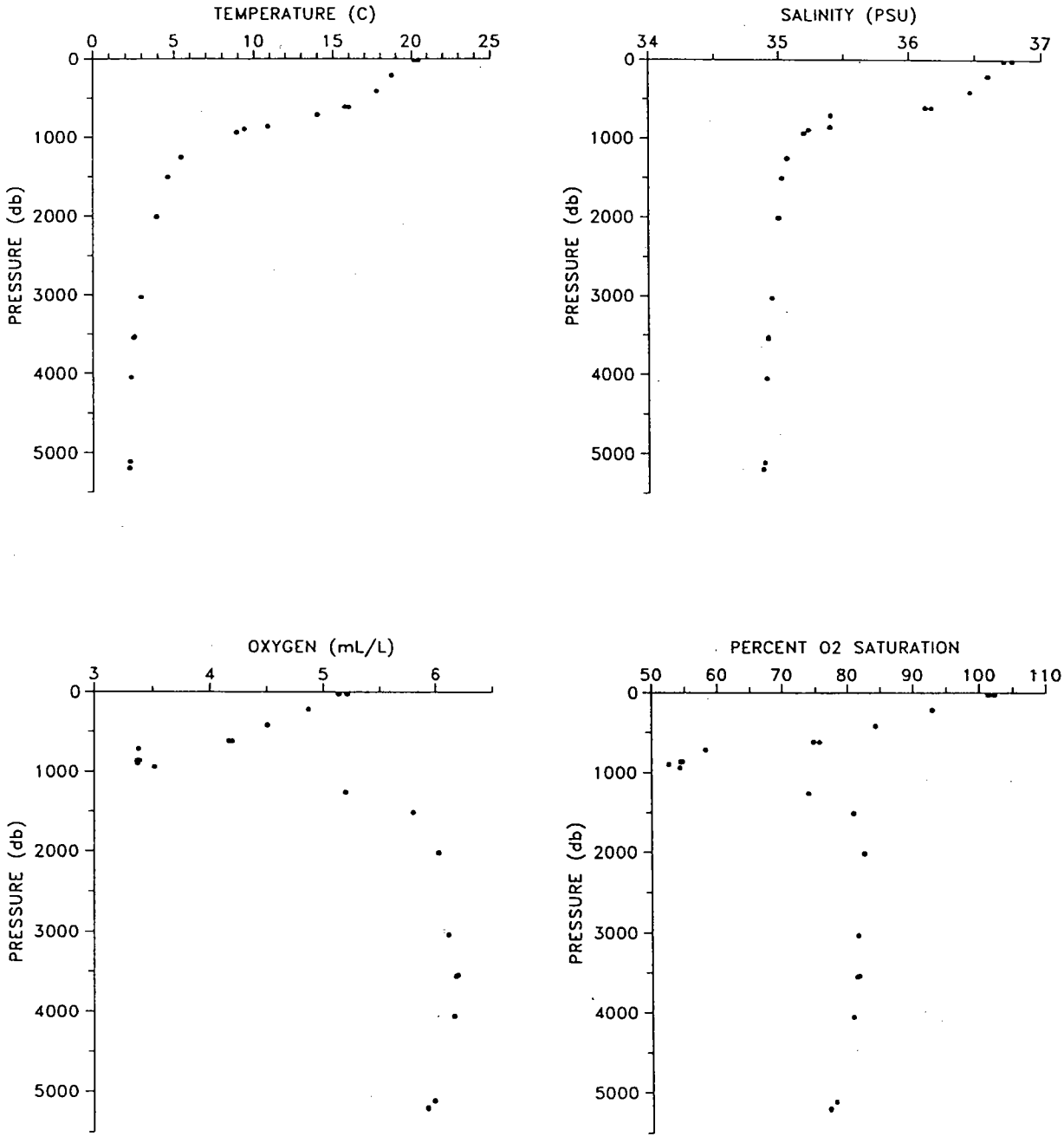

Figure 3. Hydrographic data at Station 2; casts 21, 23, and 24.

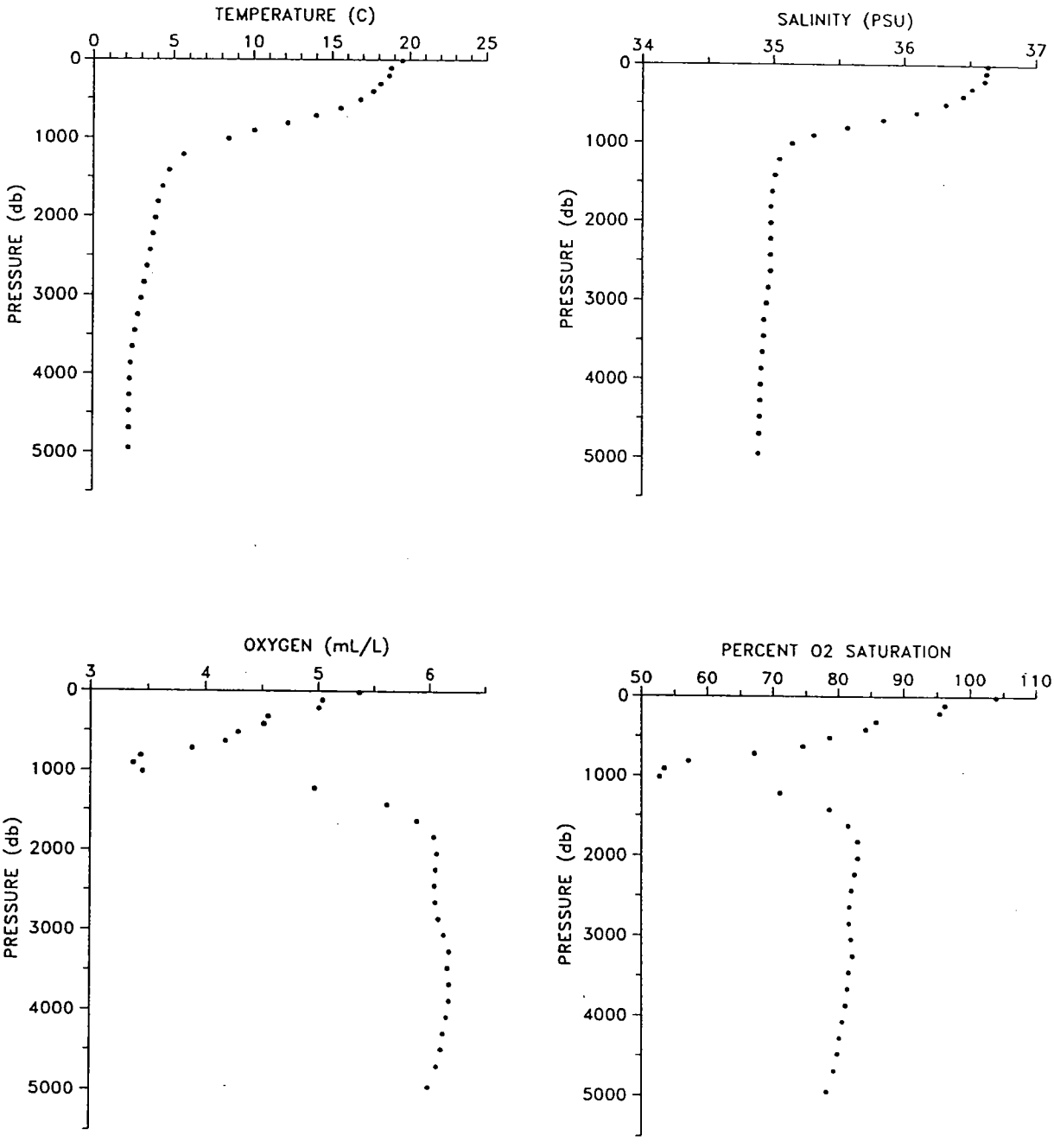

Figure 4. Bedford standards run during leg 3 of R/V Oceanus cruise 219.

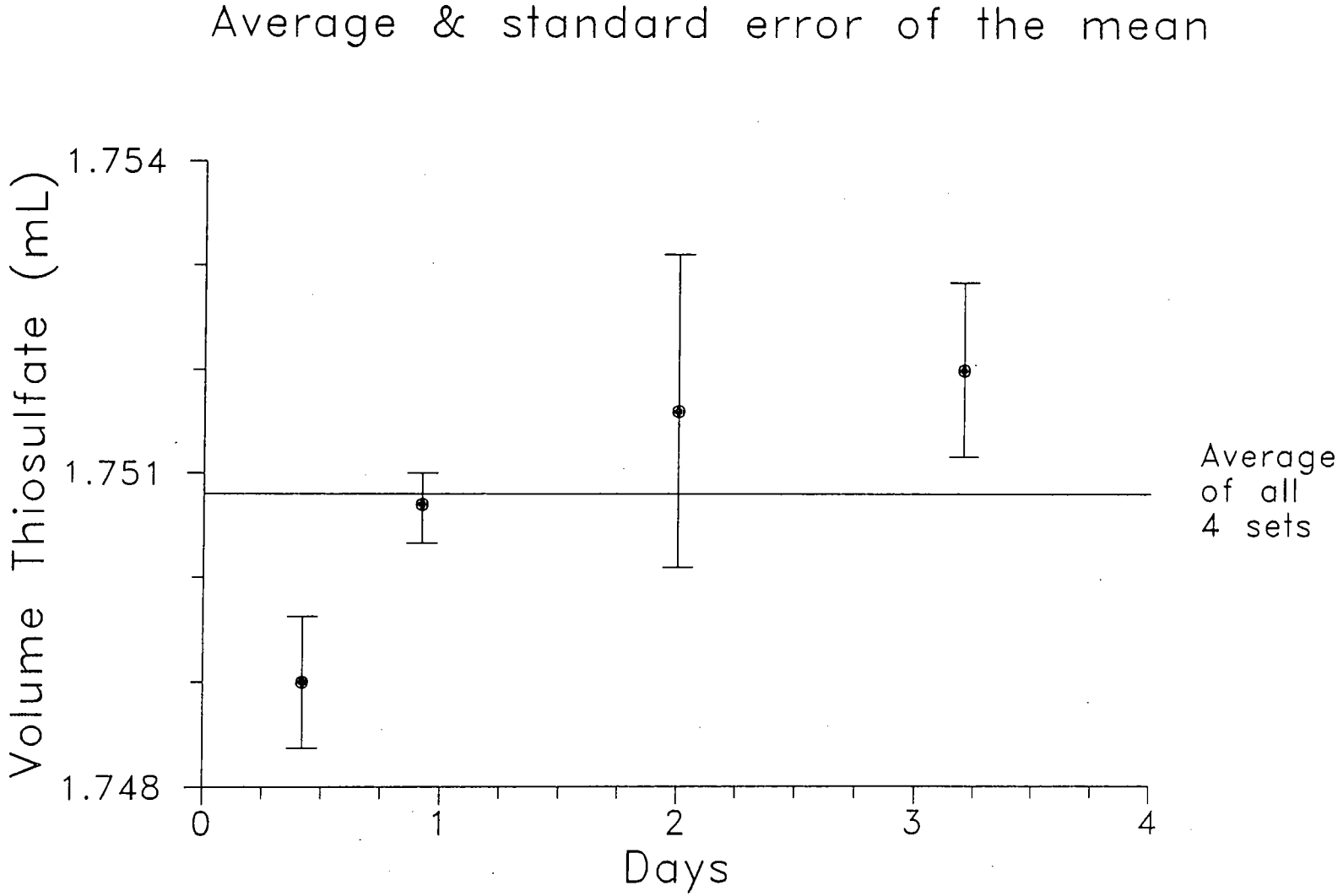

Figure 5. Delaware standards run during leg 3 of R/V Oceanus cruise 219.

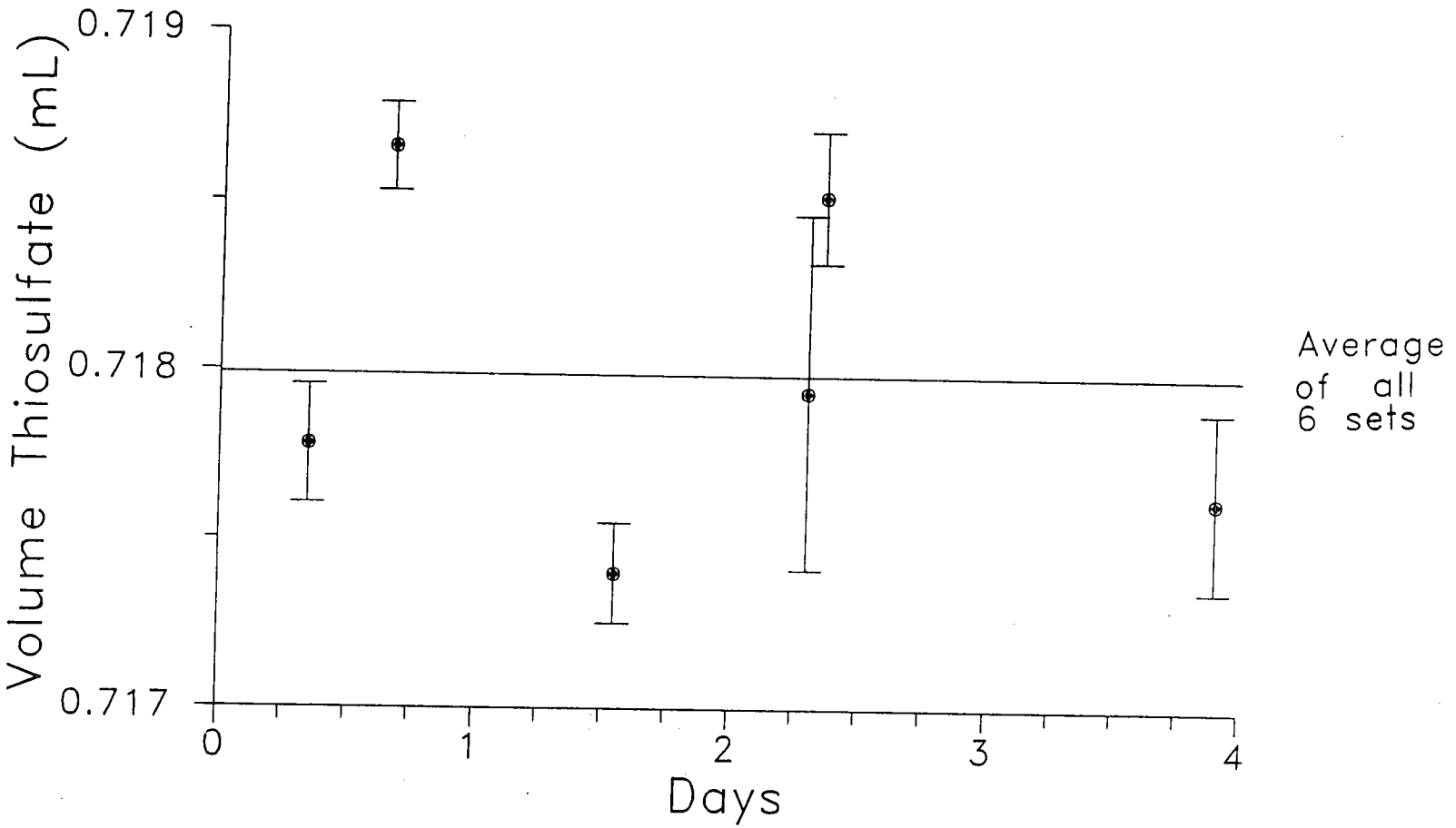

Figure 6. Scripps standards run during leg 3 of R/V Oceanus cruise 219.

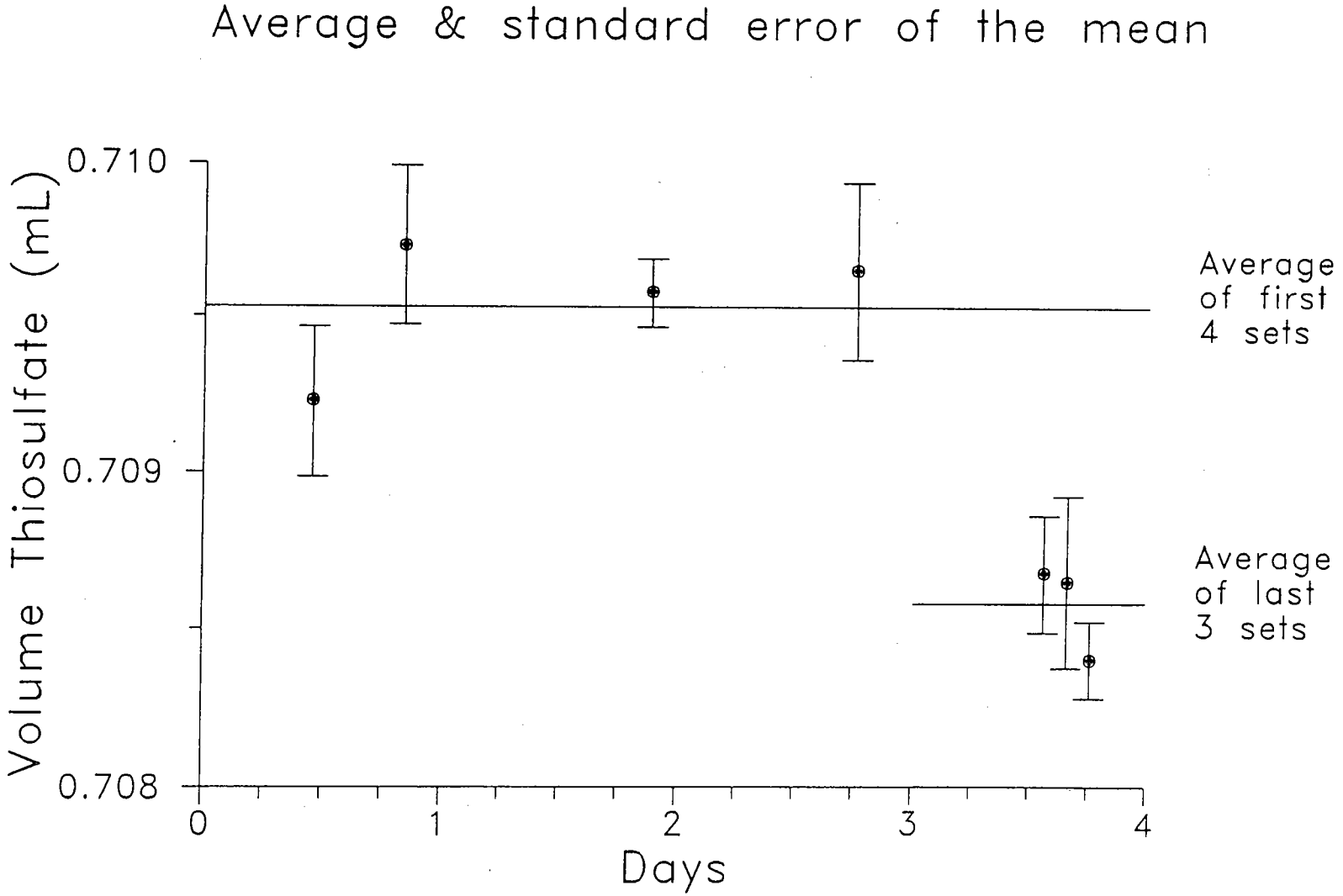

Figure 7. Woods Hole standards run during leg 3 of R/V Oceanus cruise 219.

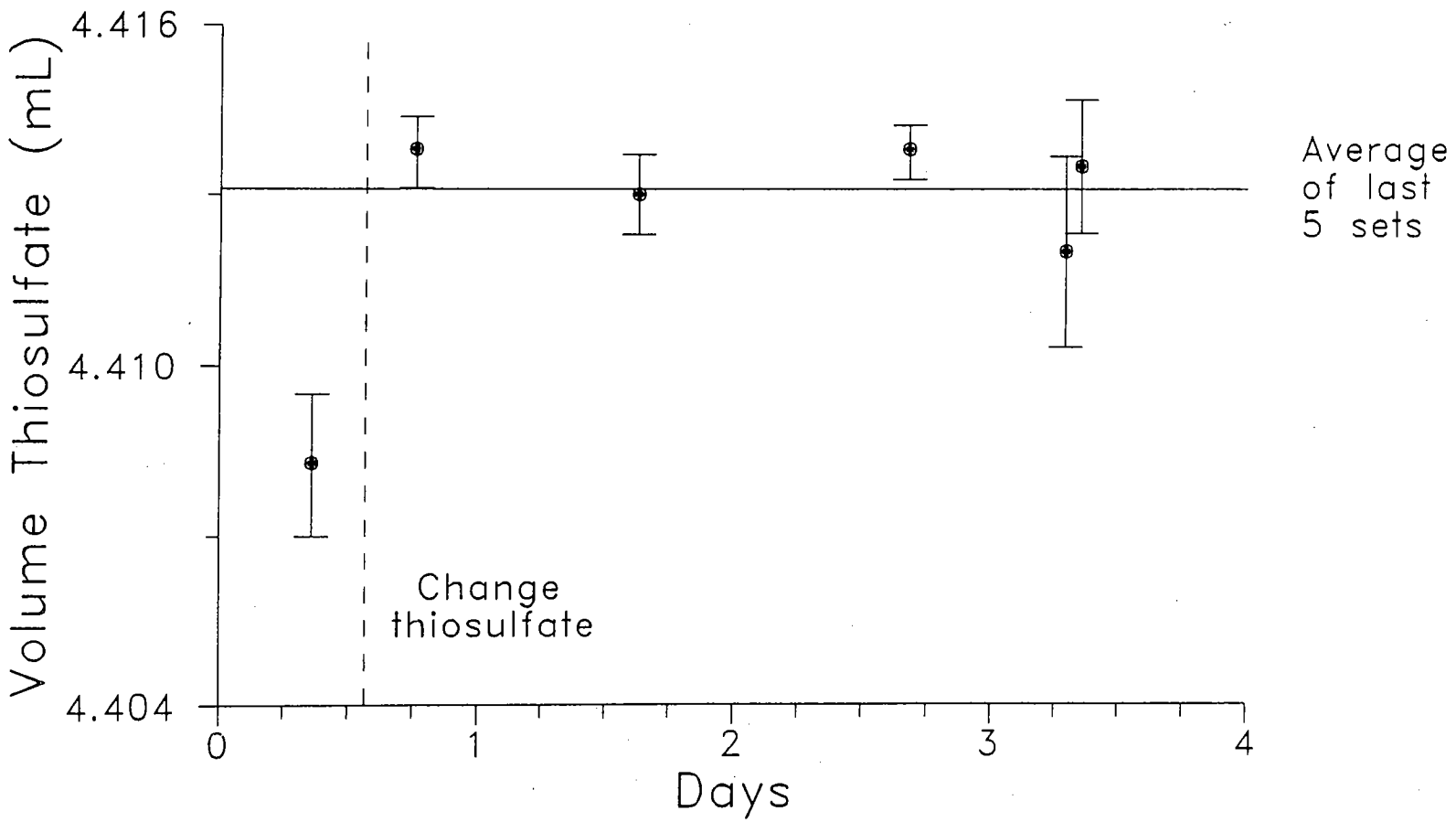

Figure 8. Bedford differences between duplicates (first titration minus second).

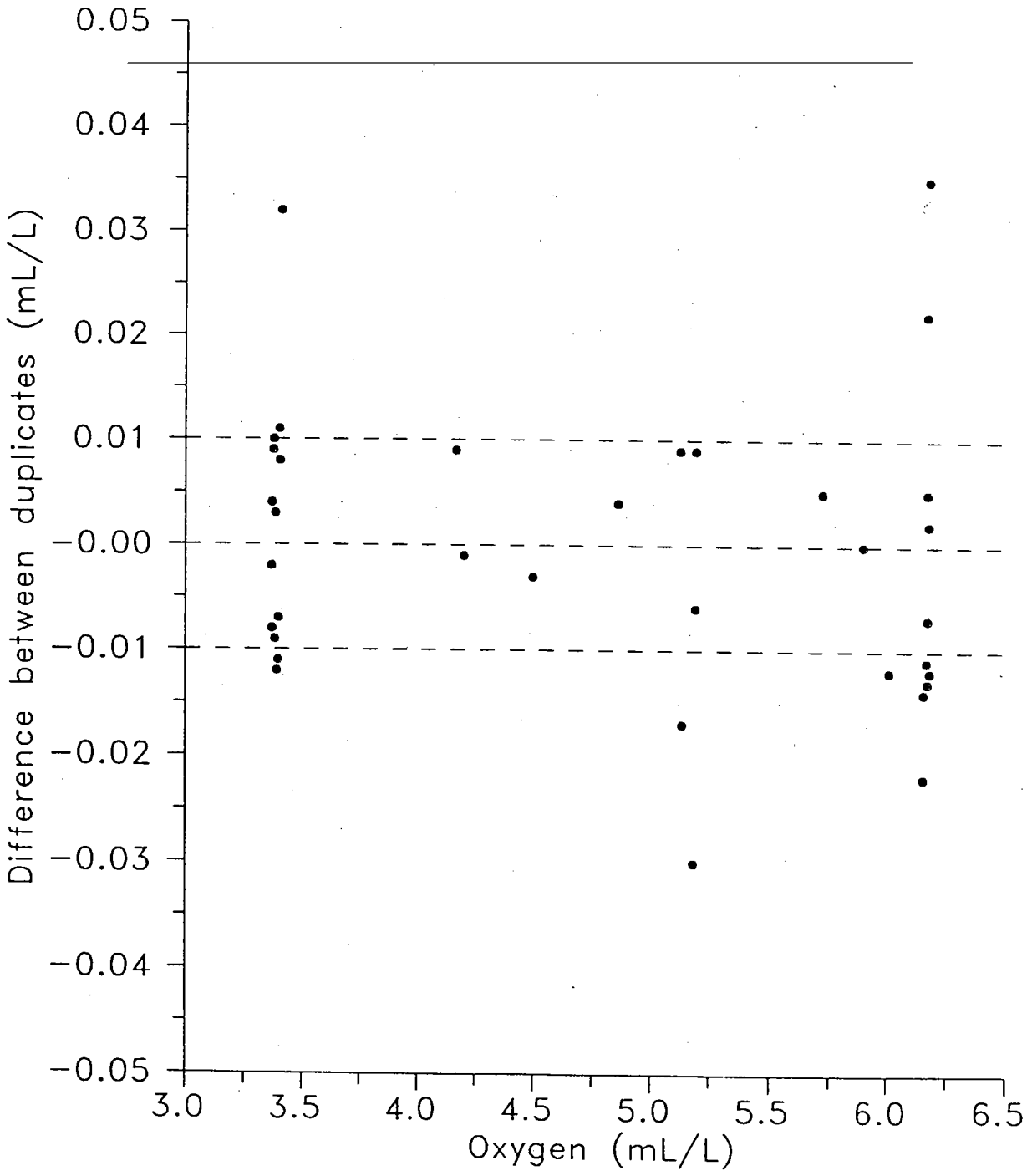

Figure 9. Delaware differences between duplicates (first titration minus second).

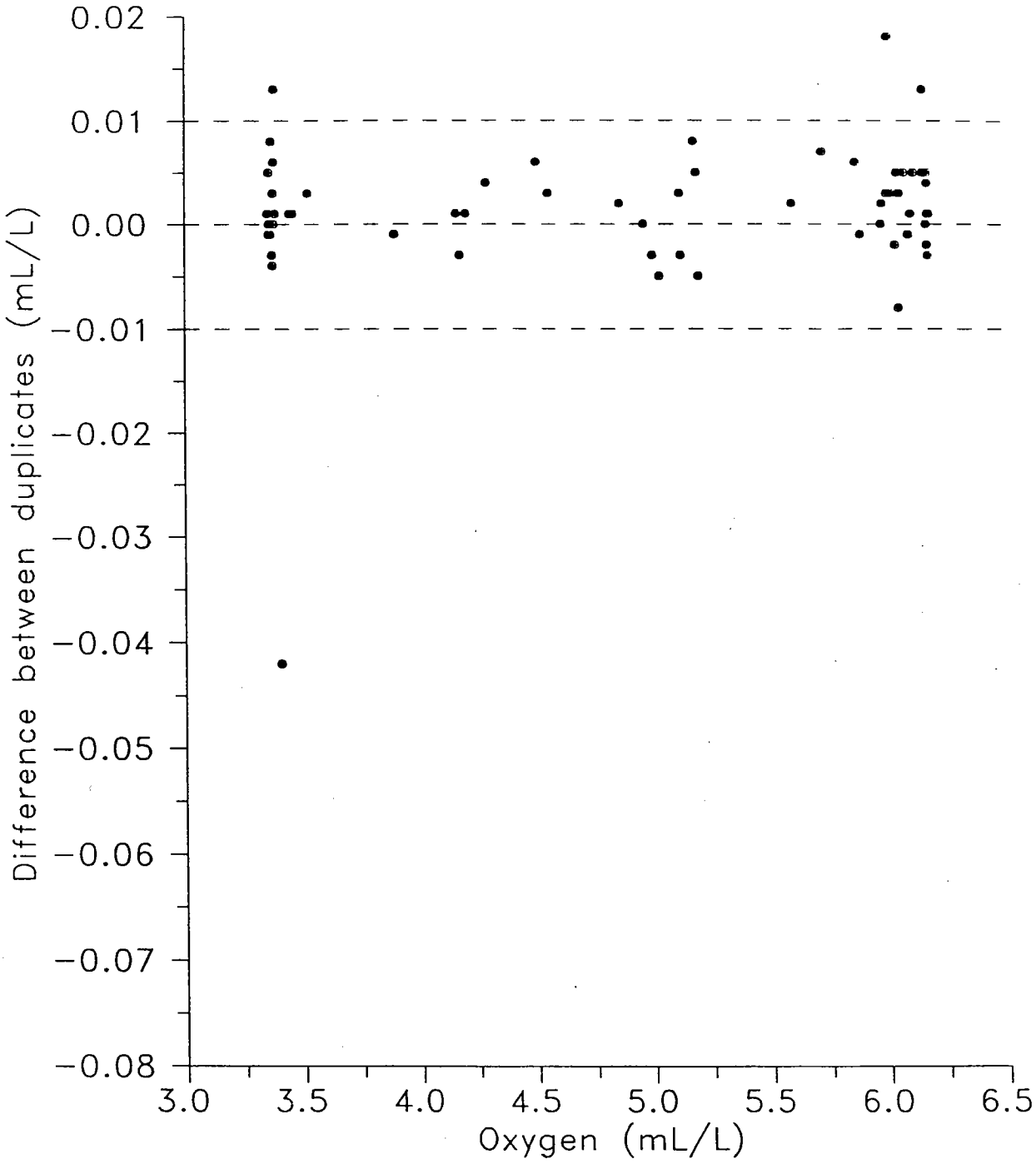

Figure 10. Scripps differences between duplicates (first titration minus second).

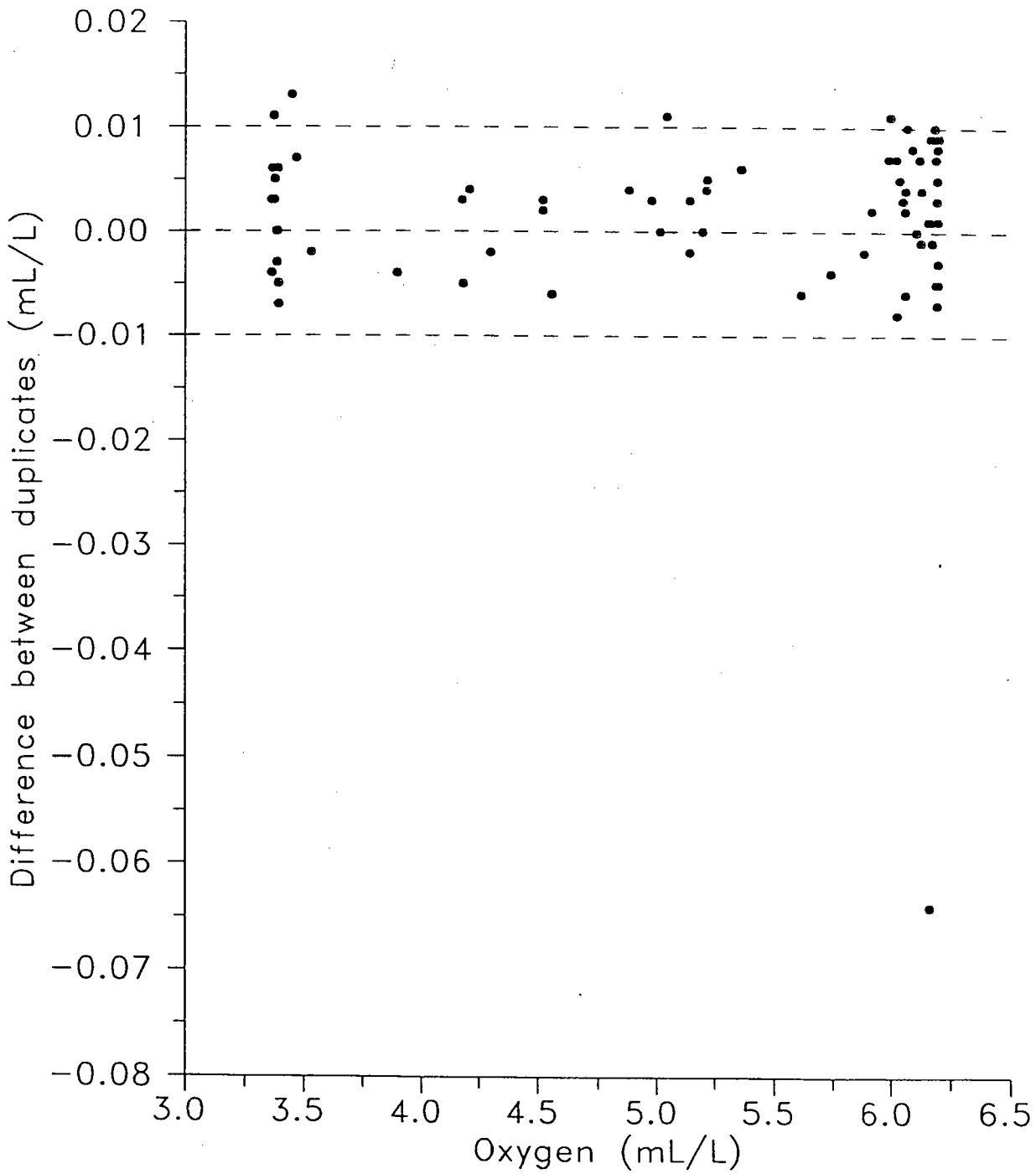

Figure 11. Woods Hole differences between duplicates (first titration minus second).

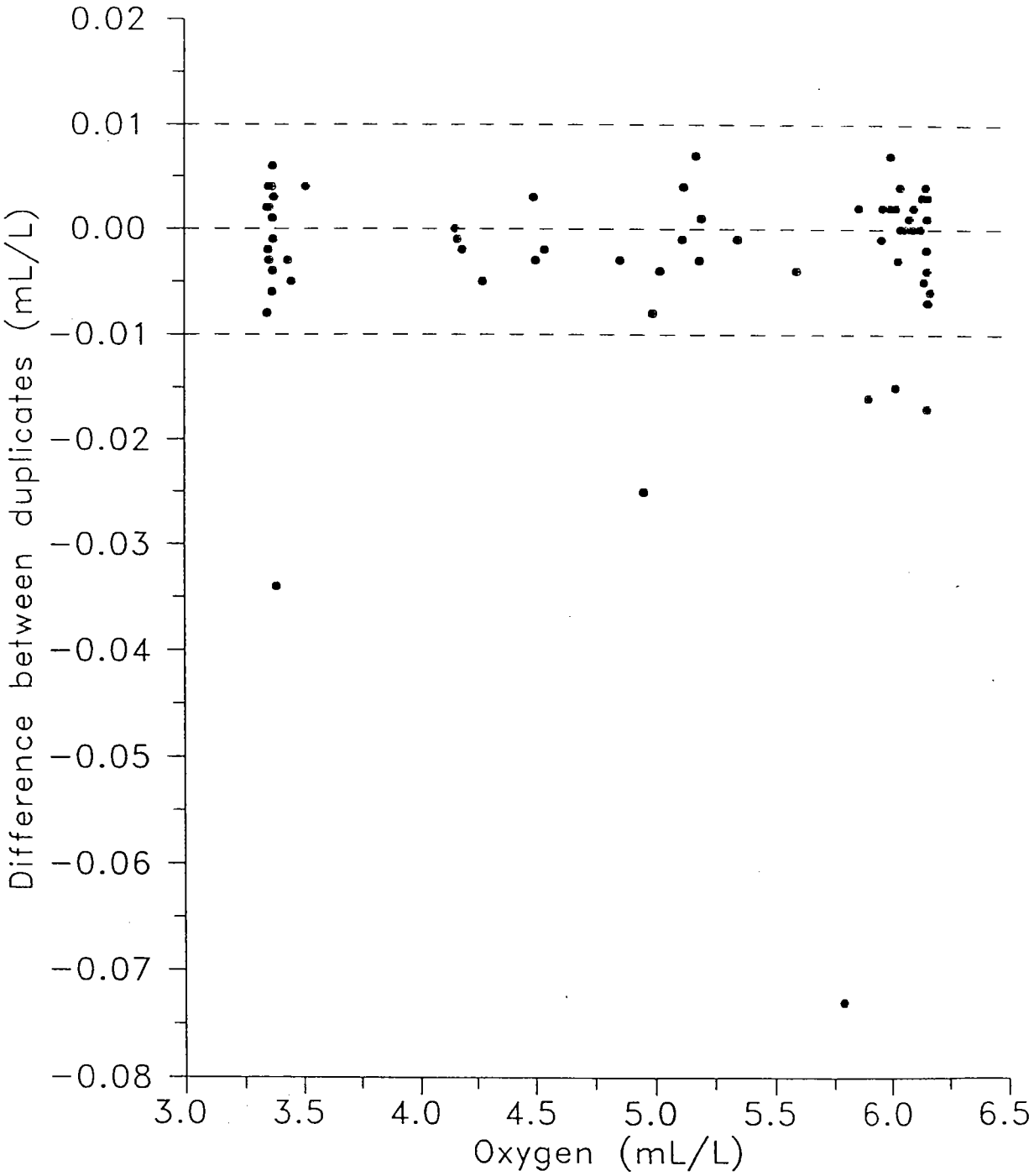

Figure 12. Bedford distribution of differences between duplicates (first titration minus second).

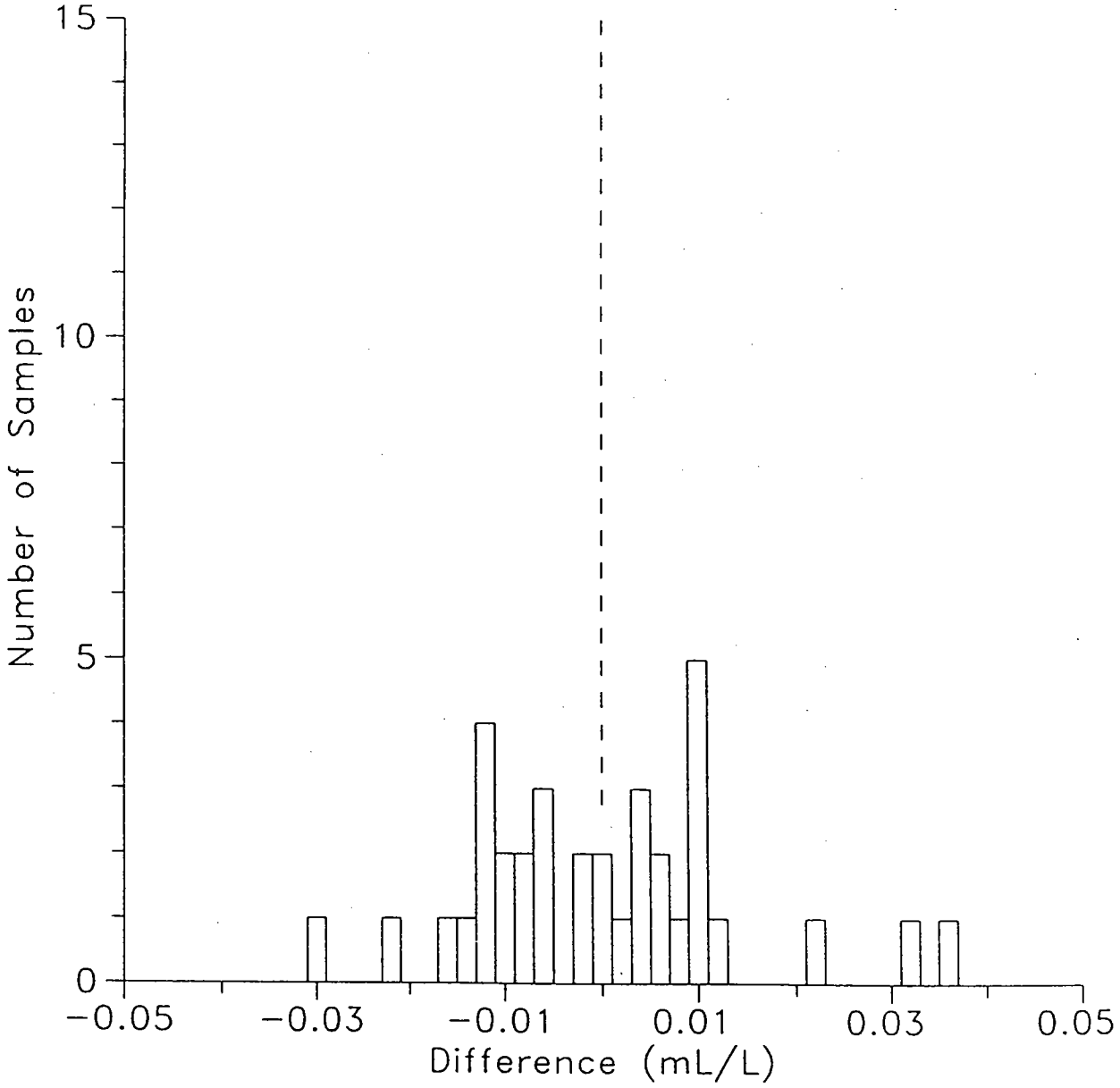

Figure 13. Delaware distribution of differences between duplicates (first titration minus second).

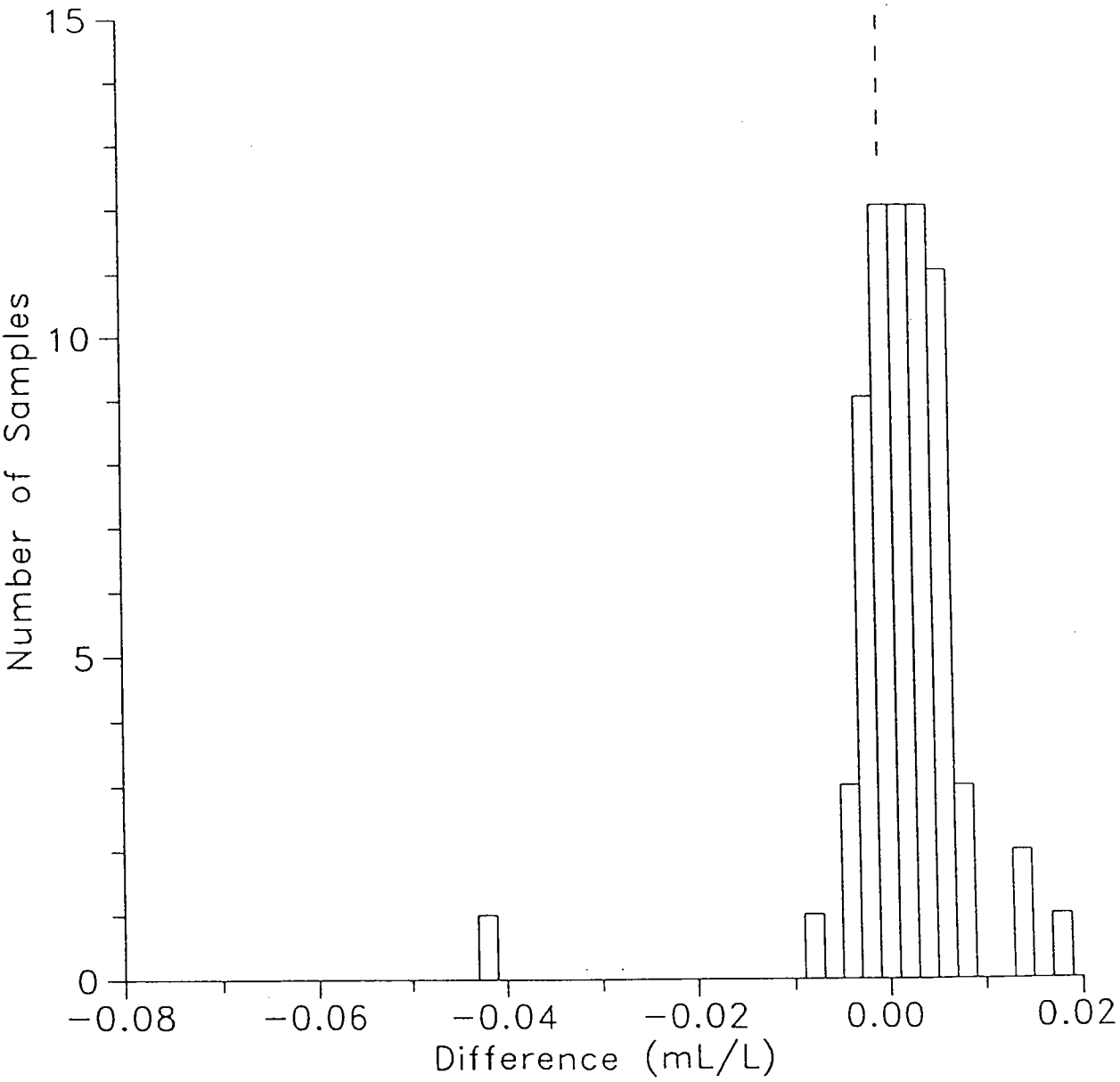

Figure 14. Scripps distribution of differences between duplicates (first titration minus second).

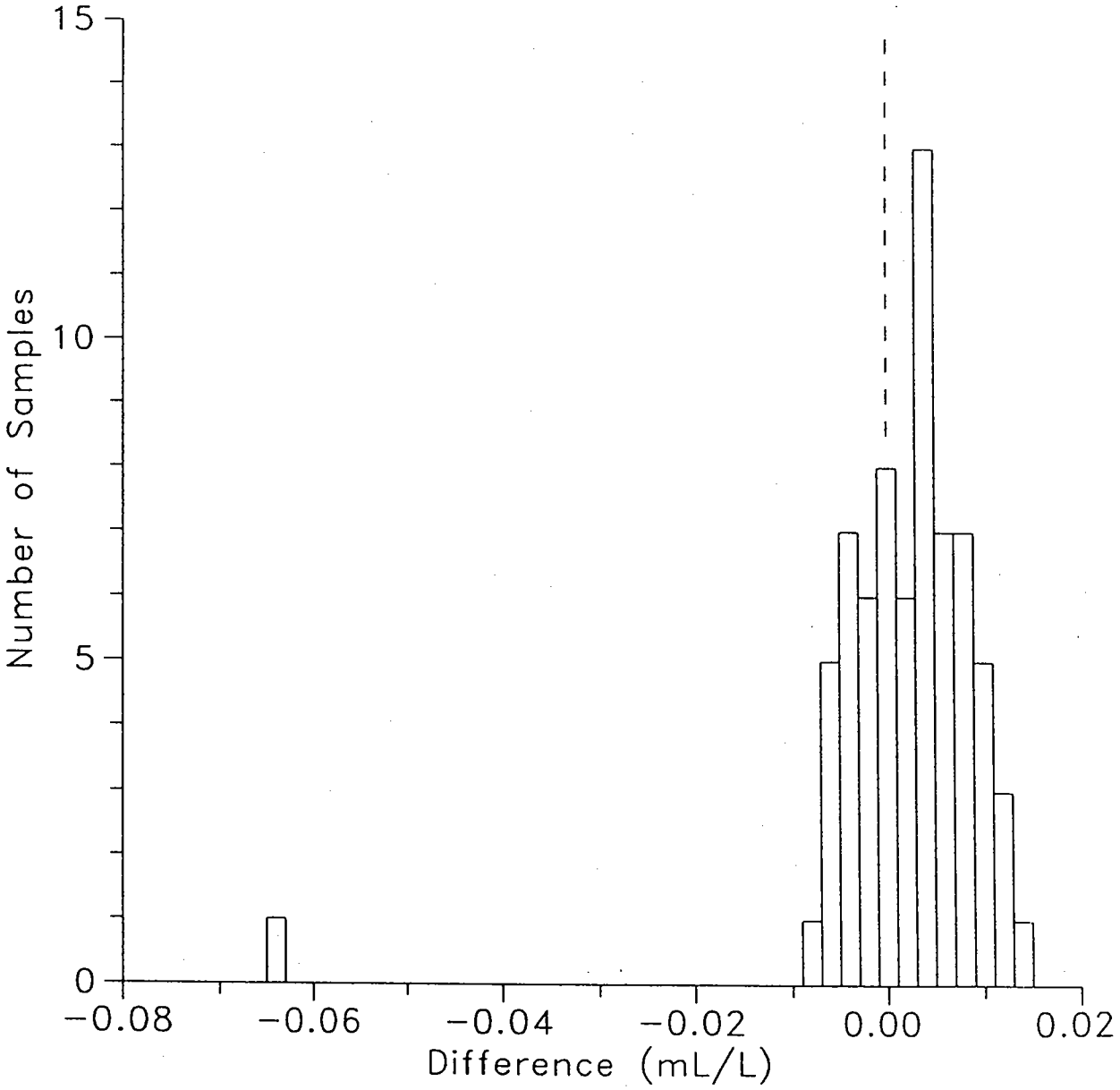

Figure 15. Woods Hole distribution of differences between duplicates (first titration minus second).

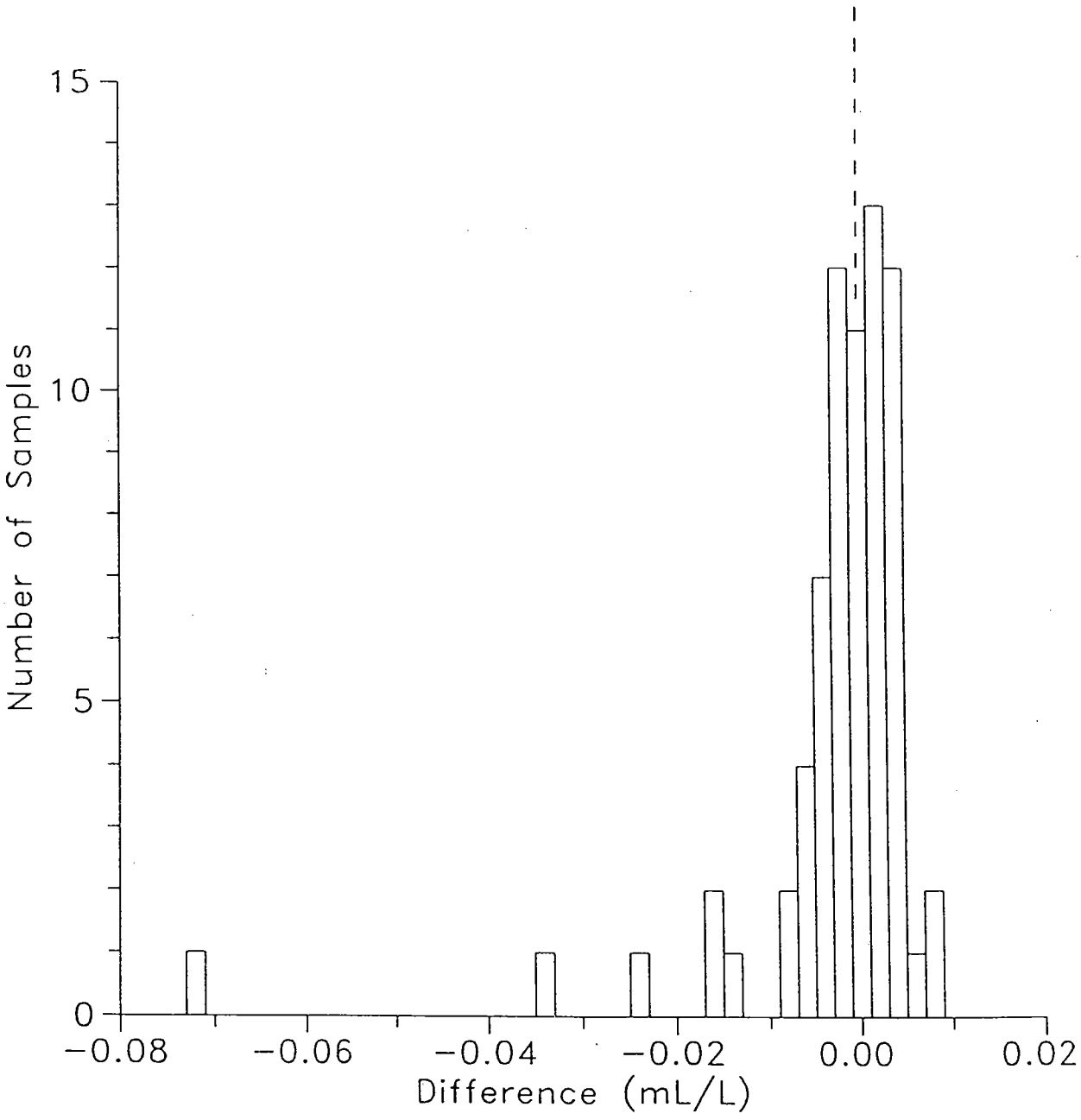

Figure 16. Difference between oxygen concentrations measured by Bedford and Delaware versus the absolute oxygen concentration.

Line: Delta O2 = −0.035 + 0.0054*O2

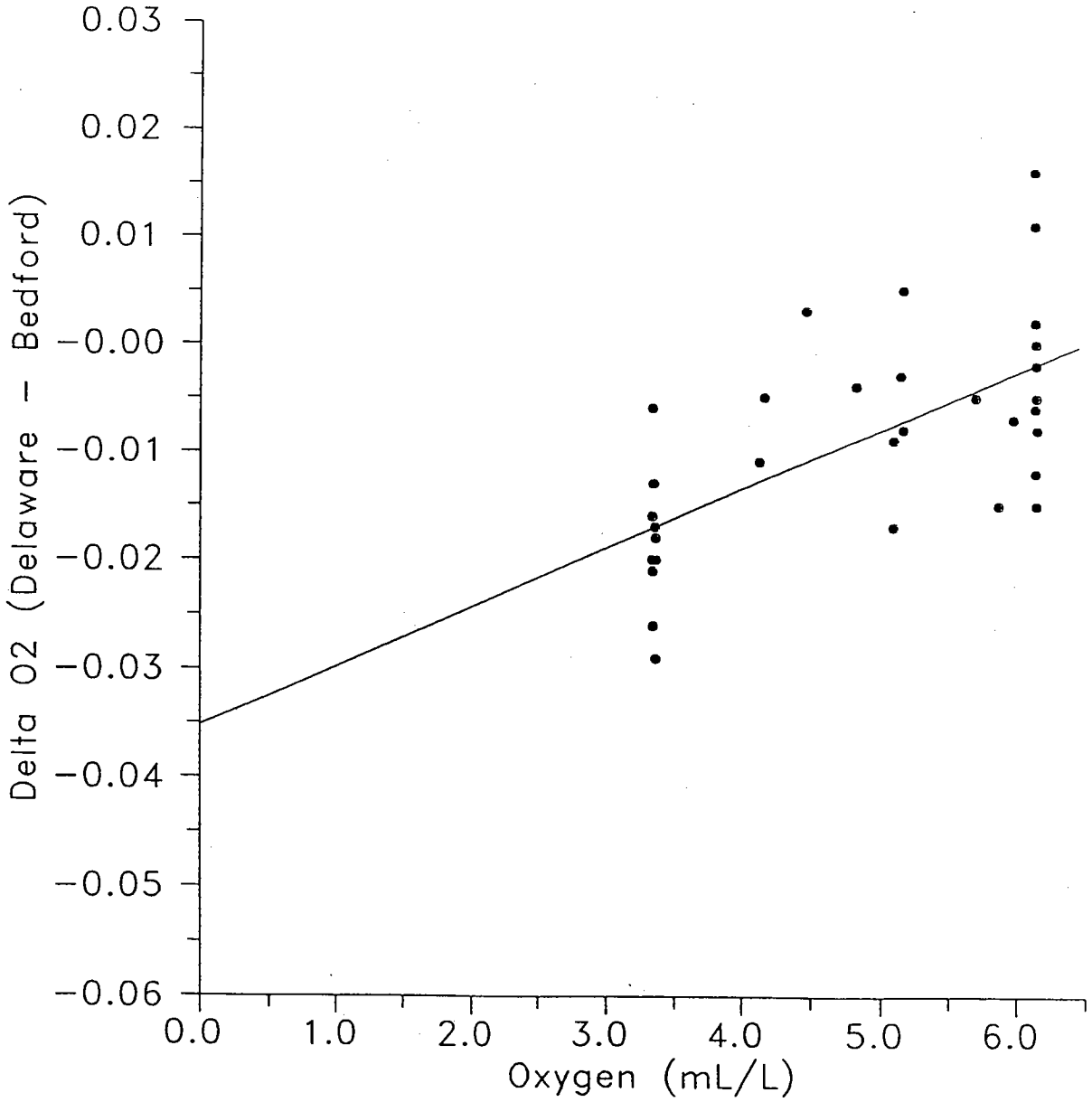

Figure 17. Difference between oxygen concentrations measured by Bedford and Scripps versus the absolute oxygen concentration.

Line: Delta O2 = −0.041 + 0.0113*O2

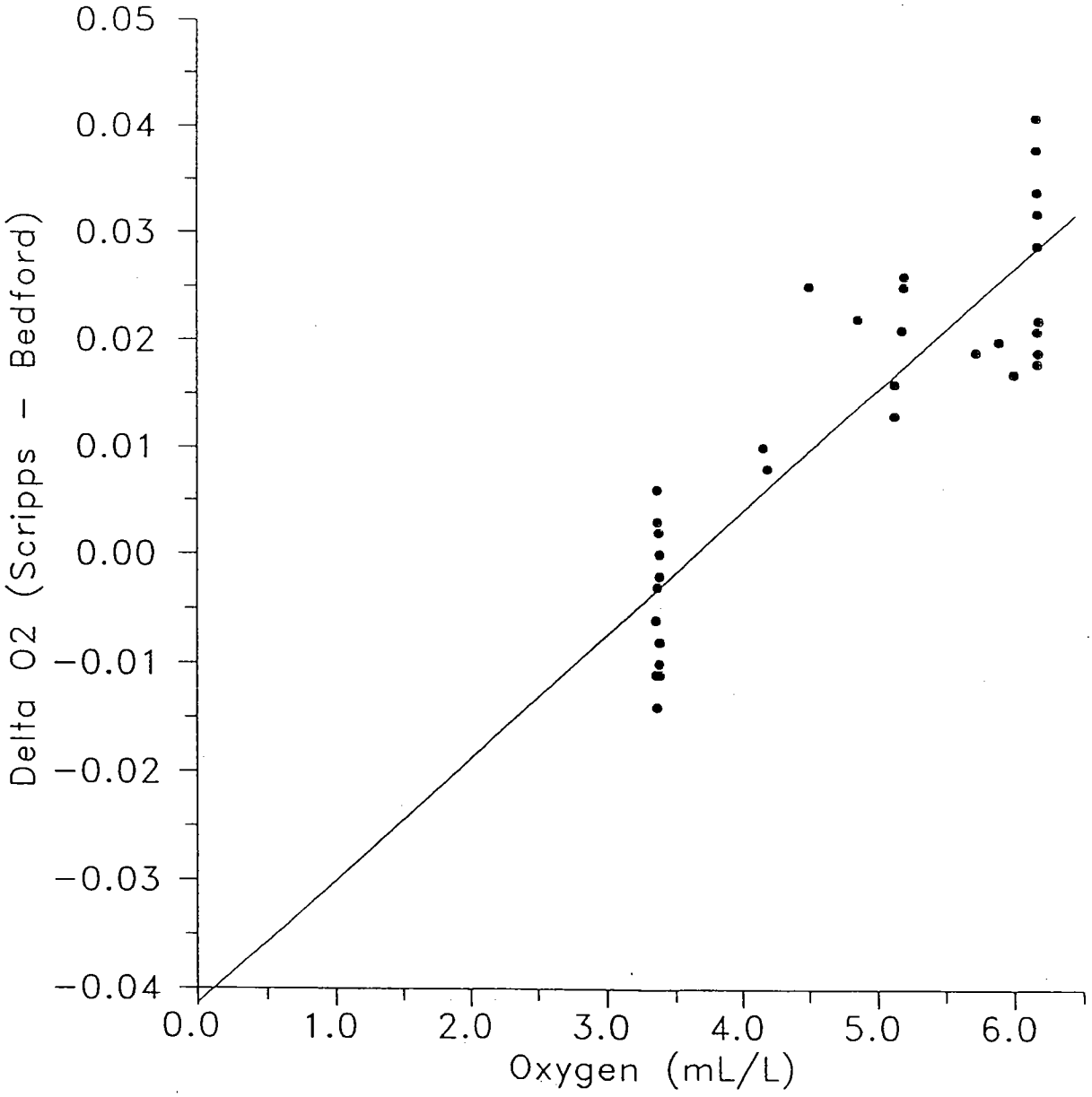

Figure 18. Difference between oxygen concentrations measured by Bedford and Woods Hole versus the absolute oxygen concentration.

Line: Delta O2 = −0.051 + 0.0092*O2

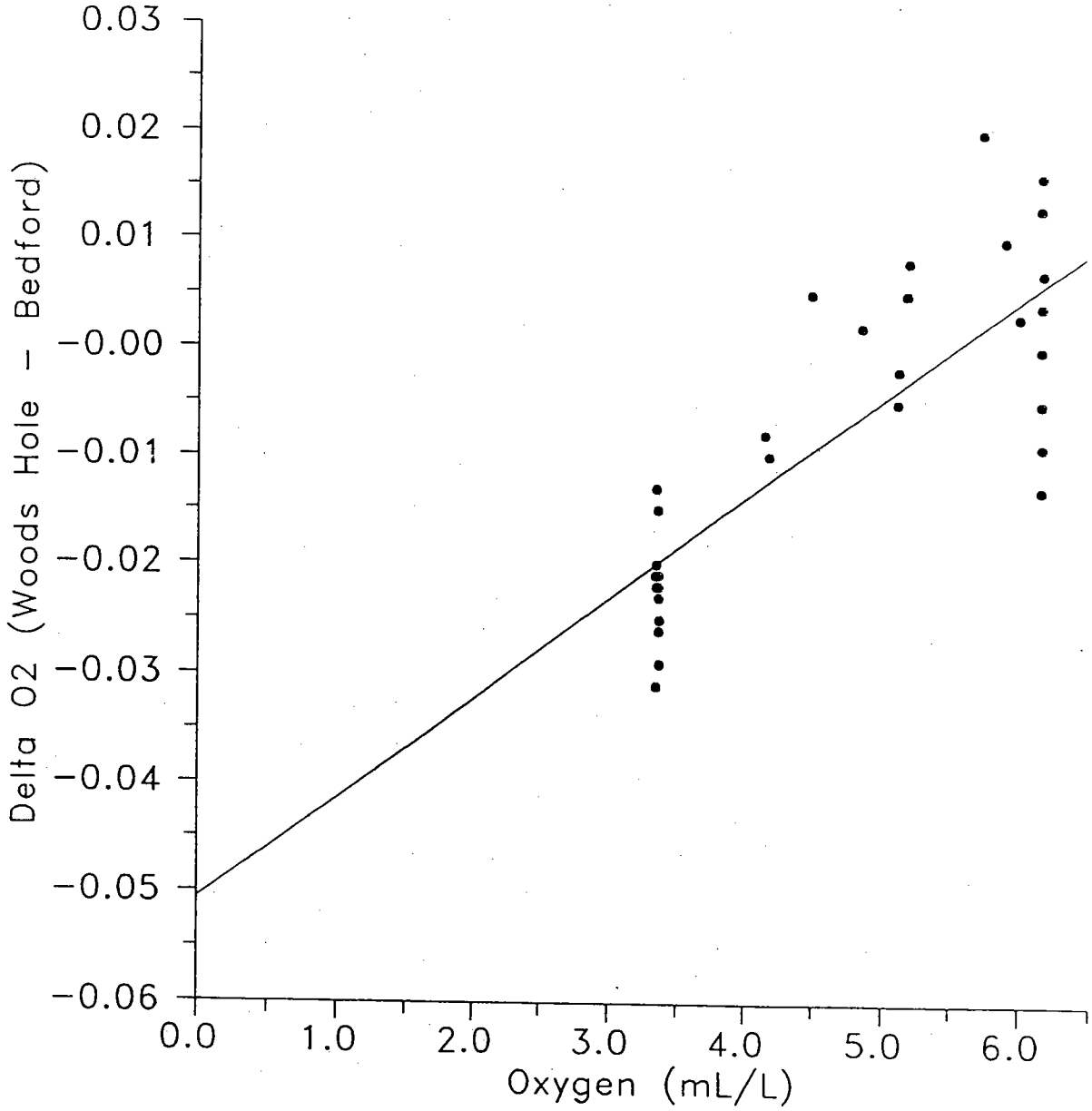

Figure 19. Difference between oxygen concentrations measured by Scripps and Delaware versus the absolute oxygen concentration.

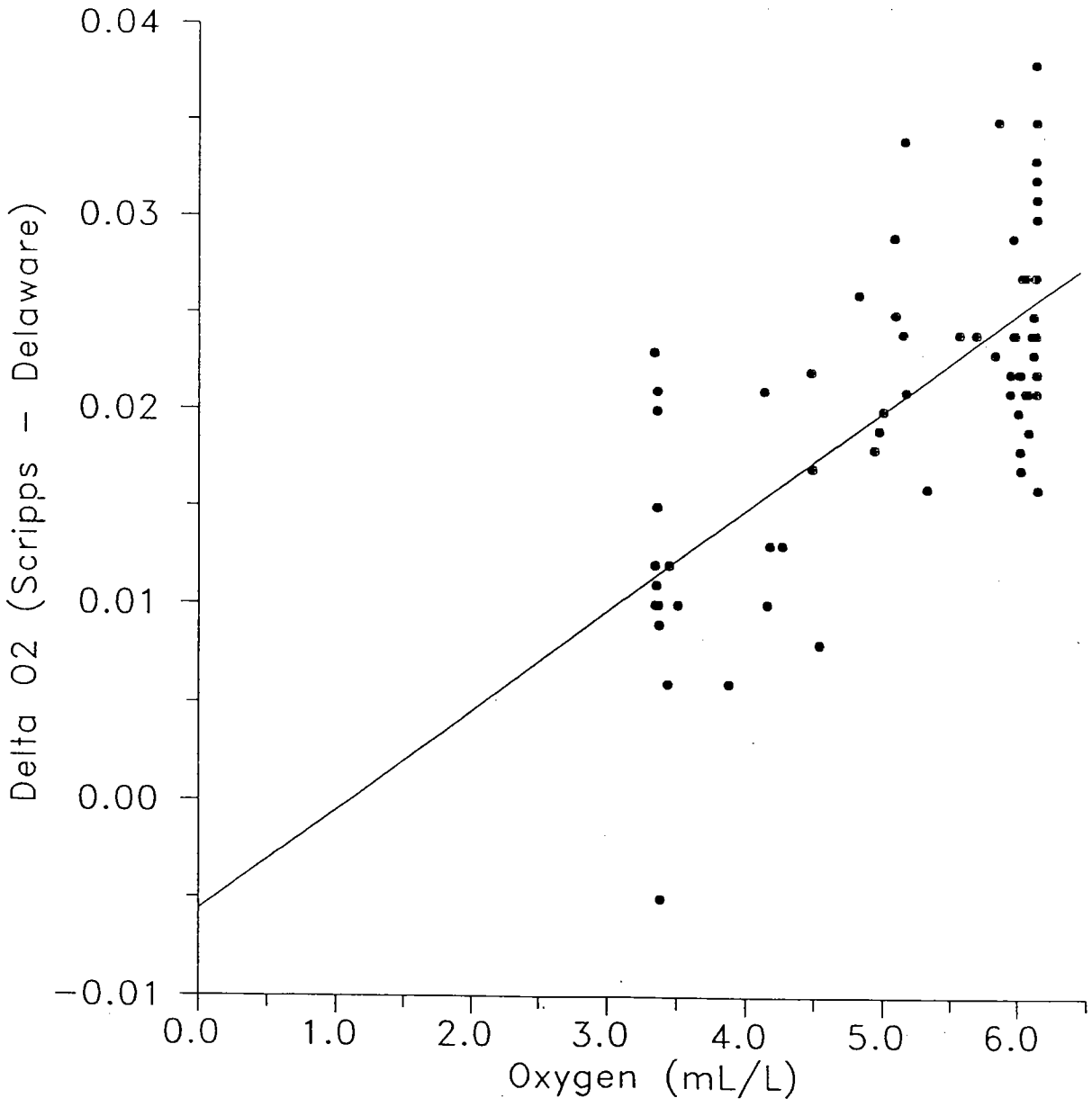

Figure 20. Difference between oxygen concentrations measured by Woods Hole and Delaware versus the absolute oxygen concentration.

Line: Delta O2 = −0.015 + 0.0032*O2

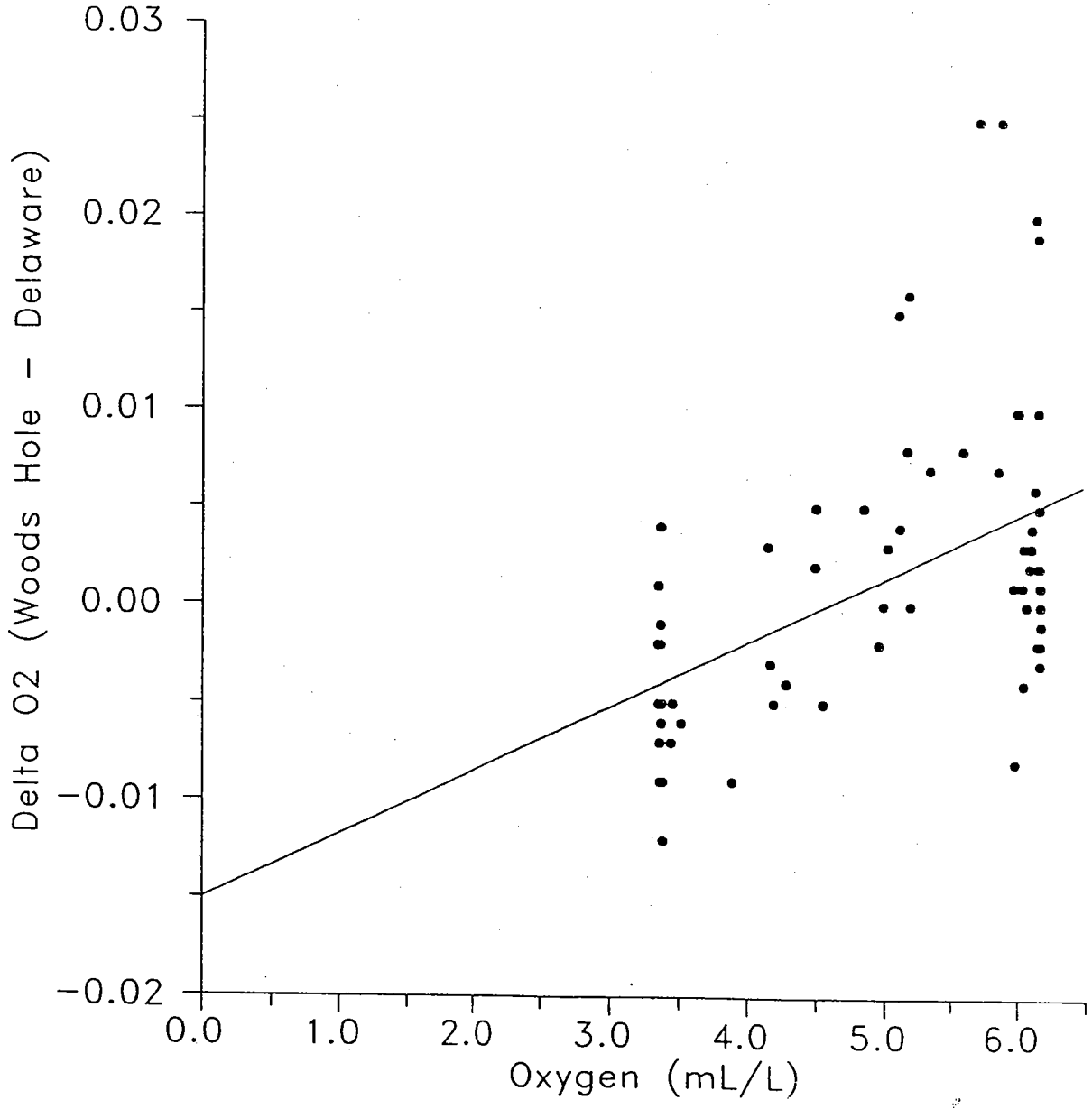

Figure 21. Difference between oxygen concentrations measured by Scripps and Woods Hole versus the absolute oxygen concentration.

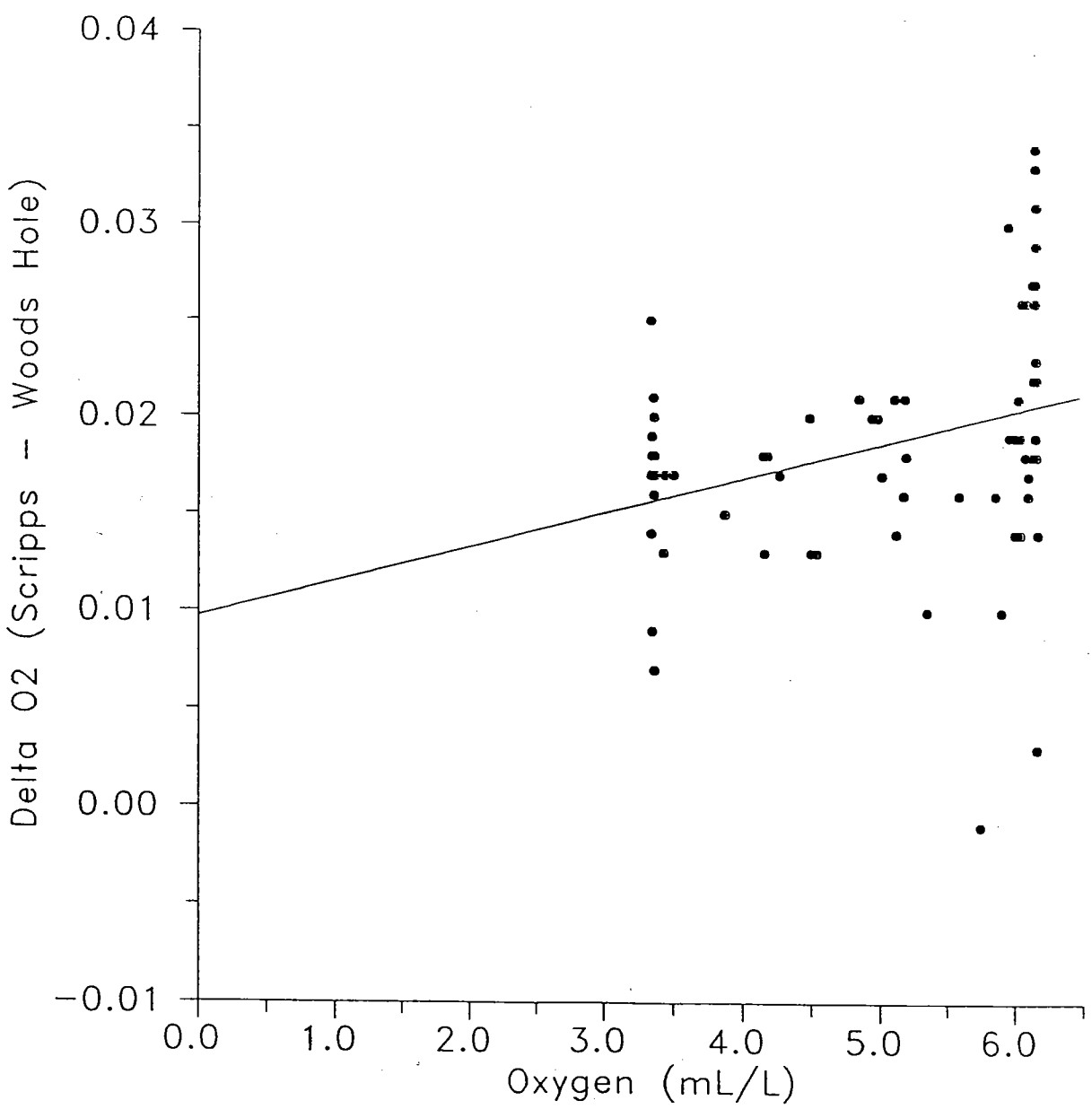

Figure 22. Comparison of regression lines: difference in concentration between institutions versus concentration. Diagonal dashed lines represent errors of ±0.5 percent.

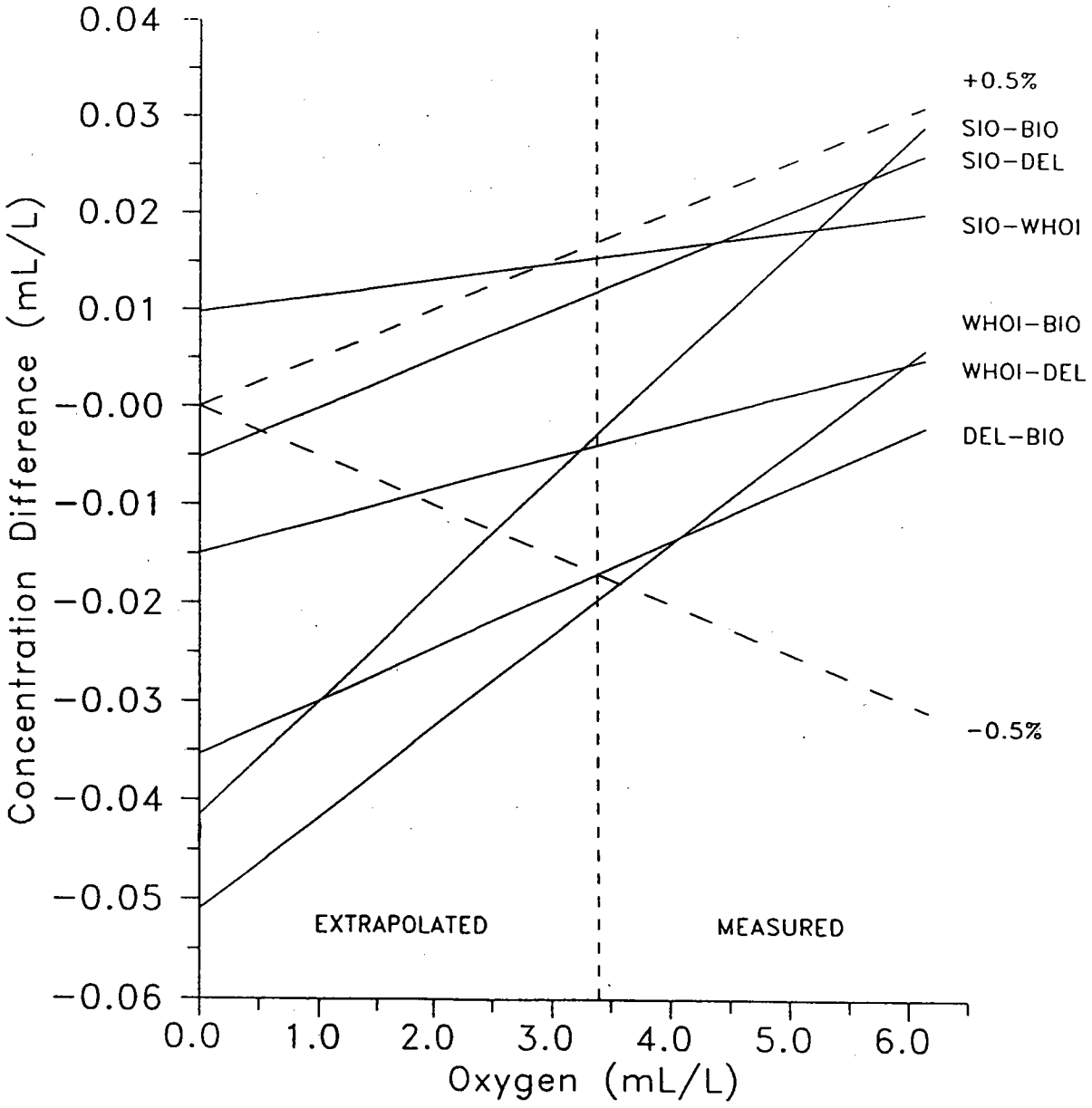

Figure 23. Temperature increase in 10 L Niskin bottles. Solid circles, temperature when bottle first opened minus potential temperature. Open circles, temperature after last sample drawn minus potential temperature.

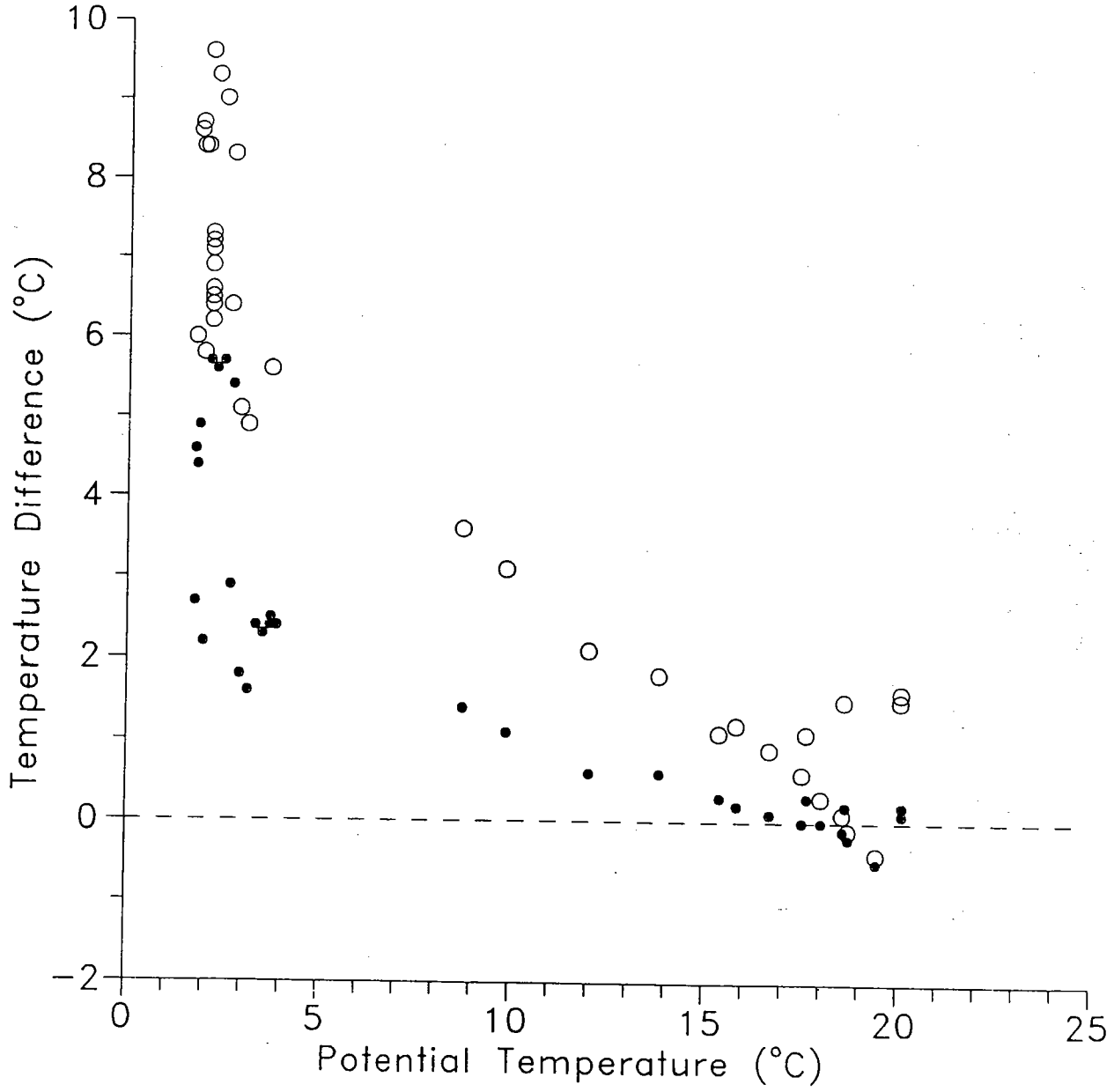

Figure 24. Temperature increase in 10 L Niskin bottles after bottles were brought on deck. Start temperature is the temperature just before first sample was drawn, end temperature is the temperature after last sample was drawn.