

COOPERATIVE RESEARCH REPORT

No. 67

THE INTERNATIONAL INTERCALIBRATION
EXERCISE FOR NUTRIENT METHODS

Organized under the auspices of
ICES/SCOR

by the

ICES Working Group on Chemical Analysis of Seawater

Edited by

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INTRODUCTION

This report is concerned with the final statistical evaluation of the data obtained during the International Intercalibration Exercise organized by the ICES Working Group on Chemical Analysis of Seawater. Before coming to the specific details of this exercise it may be useful to review briefly the history of this exercise and the specific reasons why it was carried out.

Intercalibration of chemical methods for nutrient analysis has long been the concern of an active group of chemists belonging to ICES, especially those from Baltic countries. Already in 1965 an intercalibration exercise was organized in Copenhagen between institutes in Gothenburg, Helsinki and Kiel. The results were published in UNESCO technical papers in marine science No 3. The same year a Working Group on intercalibration and standardization of chemical methods (to-day W.G. on Chemical Analysis of Seawater) was established (C.Res. 1965/33). In 1966 two intercalibration trials were performed i) In Leningrad in connection with the 5th Conference of Baltic Oceanographers and ii) in Copenhagen during the Annual Meeting of ICES. Again, the results were published by UNESCO as Rep. No 9. In general, these exercises indicated that, especially in areas where many Institutions contribute to the basic data bank, intercalibration of methods is of utmost importance. In addition, especially in multi-ship surveys, it will be worthwhile to pay close attention to a careful intercalibration of methods, either by means of stored samples or by means of a reference station.

In order to draw attention to the intercalibration problem, SCOR 1968 suggested that a world-wide international intercalibration exercise should be organized by the Working Group. The request was accepted (C.Res. 1968/3:4) and Drs. F. Koroleff and K.H. Palmork were nominated principal organizers. Subsequently, a SCOR-sponsored meeting was held in La Jolla, in which Drs. K. Sugawara, N. Rakestraw, W. Wooster and J. Gieskes participated. At this meeting, it was recommended that standards for nitrate, nitrite, phosphate and silicate would be prepared by the Analytical Chemistry Section, Sagami Chemical Research Center, under the supervision of Professor Ken Sugawara. The collection and statistical analysis of the data would then be the responsibility of Drs. Koroleff and Palmork.

The invitation to take part in the calibration exercise was sent by circular letter from SCOR to national committees, by information at ICES and other meetings and by personal letters. At the end of 1969 a positive response was received from 55 laboratories. The samples were distributed from the Sagami Chemical Research Center at the turn of the years 1969-70. Finally 45 laboratories participated in the intercalibration.

Two progress reports (C:33 and C:21) have been presented at the statutory meetings of ICES in 1970 and 1972, after which a statistical evaluation of the data was made which, due to several inadvertent delays, was finished in April, 1976. Although it has taken considerable time to reach this final end product, it seems very worthwhile to carefully consider the results of this exercise. It has been established clearly that intercalibration of methods is of great importance, that uniformly acceptable standards should be established and that, especially for open ocean surveys, the use of standard reference stations should be encouraged for the purpose of in situ intercalibration.

Participating laboratories

This world wide intercalibration of nutrients was joined by 45 laboratories from 20 different countries (see list of participants). Each laboratory was given a randomly picked number from 1 to 46 (number 13 was omitted).

List of participants

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DESCRIPTION OF THE STUDY

(1) Sample Preparation

All standards were prepared by the Sagami Chemical Research Center under the supervision of Professor K. Sugawara. The preparation and validity of these samples has been described by Prof. Sugawara in a series of unpublished progress reports, collected in a booklet 1969. Sample kits were sent to the participants with detailed instructions on the procedure to be followed to complete all the nutrients tests.

Briefly, the suggested procedure was as follows:

1) P-solutions.

a. Blank solution

Take the P-blank ampoule. Stir and cut the narrow neck. Transfer a necessary volume of the contents to a receiver. Add necessary amounts of reagents. Determine the transmittance or absorbance of the solution. The test is repeated two more times by using the remaining solution.

b. Take the P-low solution and process it as with blank solution above, to give three series of data for the P-low solution.

c. The same test is conducted by using the P-medium and P-high ampoules.

2) Si-solutions

a. Blank solution

Take one polyethylene Si blank bottle. Remove outer and inner lids. Break the parafilm seal if necessary. Pipet a necessary volume to a receiver to be treated with reagents according to your own method. Then the transmittance or absorbance is determined. The test is repeated for two other blank bottles to give blank data.

- b. Three Si-low, three Si-medium and three Si-high bottles are processed in the same way successively to obtain three series of data for each of the solutions different in Si concentration.

3) Nitrite-N solutions

The nitrite-N blank solution, NO_2 -N low, NO_2 -N medium and NO_2 -N high solutions are processed by your own method to obtain three sets of data as in the case of P and Si.

4) Nitrate-N solutions

The nitrate-N solutions are processed by your own method to give series of data paralleled by other elements, data.

For completing the series of tests:

- 1 set of 4 ampoules, 0, low, medium and high in P.
- 1 set of 3 blank bottles, low, medium and high in Si each 3, totalling 12.
- 1 set of 4 bottles, 0, low, medium and high in NO_2 -N.
- 1 set of 4 bottles, 0, low, medium and high in NO_3 -N.

were sent to the participants.

(2) Analytical Methods

Phosphate

The majority of the participants used the method (M) by Murphy and Riley (1968) or a modification thereof (C). Stannous chloride (S) was used as the reduction agent in 5 experiments:

- M: A mixed reagent containing sulfuric acid, ammonium molybdate, antimony ions and ascorbic acid is used.
- C: Two reagents are used: (1) sulfuric acid, ammonium molybdate and antimony ions; (2) ascorbic acid.
- S: Two reagents are used: (1) sulfuric acid and ammonium molybdate; (2) stannous chloride.
- A: Indicates that the method is automated.

Silicate

Silicate measurements were carried out either by the measurement of the yellow silico-molybdate complex or by the reduced blue silico-molybdate complex. We denote the methods by:

- Y: Yellow silico-molybdate method.
- B: Blue reduced silico-molybdate method.

Nitrite

All laboratories used a method based on the classical Griess reaction (diazotation).

Nitrate

Various methods were used to reduce nitrate to nitrite which was then determined by the Griess reaction method. We use the following symbols:

- G: The nitrate is reduced to nitrite in an amalgamated cadmium column.
- U: The nitrate is reduced to nitrite in a copper-treated cadmium column.
- H: The nitrate is reduced by hydrazine.
- T: Determination with strychnine in concentrated sulfuric acid.

Z: Nitrate is reduced to nitrite with zinc-dust.

A: Indicates that the method is automated.

RESULTS

The data obtained in this exercise are listed in Tables 7-10. Briefly, this table lists the parameter measured, the sample package, the laboratory code, the method, the true value of the standard (in $\mu\text{g at dm}^{-3}$), the number of replicates, the mean, the variance and the observed values. Means and variances, of course, are reported to more decimals than are justified.

Since the difference in concentration between the different packages is very small, all the observed values were standardized according to a chosen standard true value for the low, medium and high concentration respectively for each parameter. The mean values from the different laboratories were then ranged from the highest to the lowest for each parameter and concentration (Table 6). In Figs. 1, 2, 3 and 4, mean values are plotted, together with the standardized true values for phosphate, nitrate, nitrite and silicate respectively. The omission of the outliers was based on the rank of the mean values from individual laboratories for the three concentrations of each parameter and the deviation of the extreme mean values from the neighbouring values (Dixon 1953). It was decided that for phosphate lab. no. 7 and 14, for nitrate lab. no. 14 and 34, and for nitrite lab. no. 6, 14 and 45 should be omitted from the further analysis on accuracy and precision.

STATISTICAL ANALYSIS

In the following, the data obtained have been analyzed statistically for the variance within and between laboratories, as well as for the precision and accuracy of the various parameters and methods.

The "within" and "between" laboratory components of the variance were estimated by the method outlined by Cochran (1963). The significance of differences between variances of different methods

were tested by assuming a F-distribution with $m-1$ and $n-1$ degrees of freedom for the ratios S_1^2/S_2^2 , where S_1^2 and S_2^2 are the estimated variances of a laboratory mean for method 1 and method 2 respectively, and m and n are the number of laboratory mean values the estimated variance is based upon (Mood and Graybill, 1963). Average deviations of the measured concentrations from true values were tested by a "Student's" t-test (Mood and Graybill, 1963).

(1) The variance within and between laboratories

For convenience, only the laboratories giving three values for the measurements of one concentration were used. (Most of the laboratories gave three values). We distinguish two components of the variance, the "within" and "between" laboratory variances. The variance on a single laboratory mean, based on three replicate measurements, is given by

$$(1) \quad S^2 = S_b^2 + S_w^2/3$$

where S_b^2 = "between" laboratory variance

S_w^2 = "within" laboratory variance

For a given parameter, concentration and method, S^2 may be estimated from the set of laboratory means, and S_w^2 may be estimated by taking the mean of the variances within each laboratory, i.e.

$$S^2 = \sum_{i=1}^n \frac{(\bar{x}_i - \bar{x})^2}{n-1}$$

and

$$S_w^2 = \frac{1}{n} \sum_{i=1}^n \sum_{j=1}^3 \frac{(x_{i,j} - \bar{x}_i)^2}{2}$$

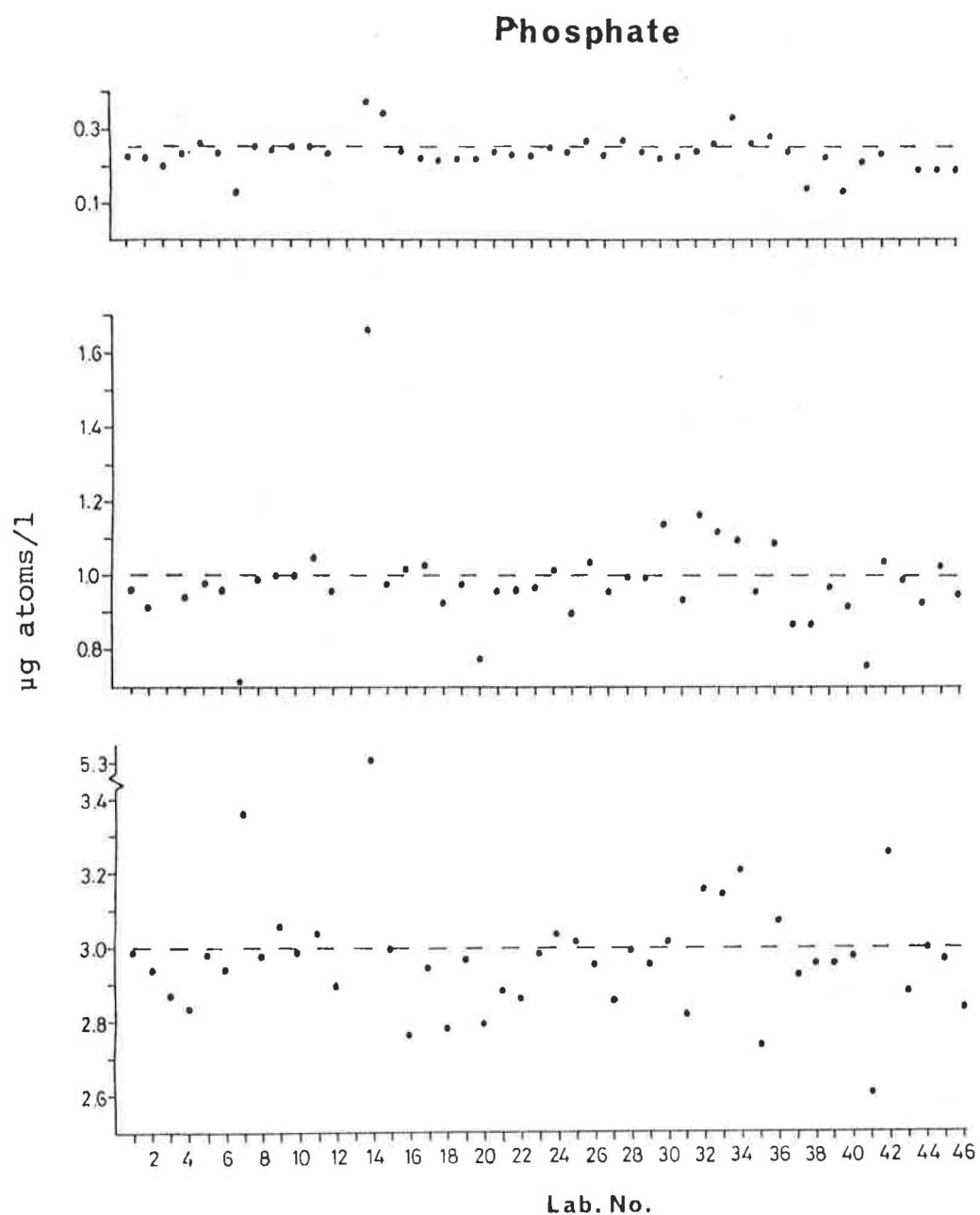


Fig.1. Standardized mean values of phosphate measurements reported by the different laboratories for the low, medium and high concentration. Dotted lines: Standardized true values.

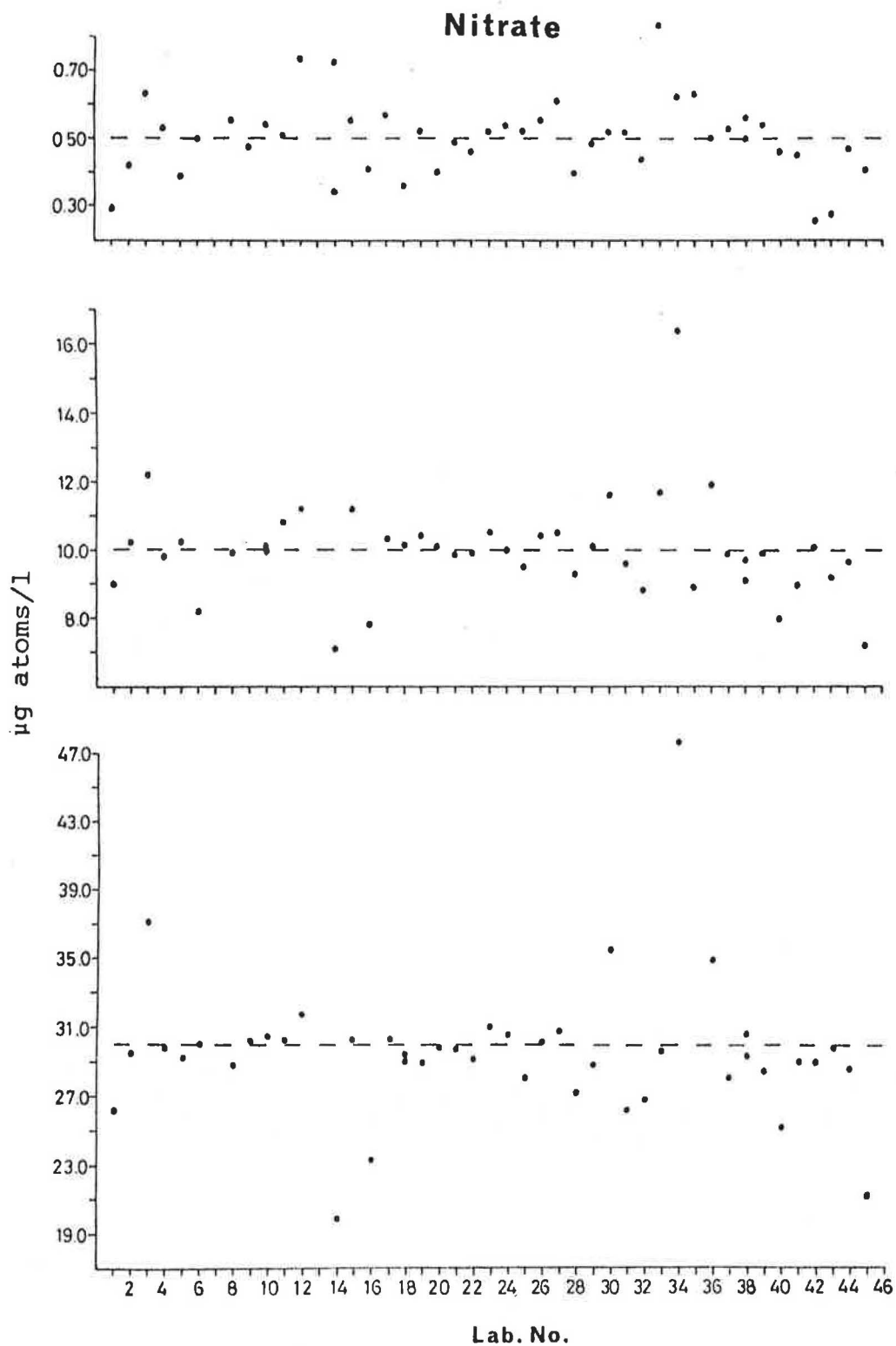


Fig.2. Standardized mean values of nitrate measurements reported by the different laboratories for the low, medium and high concentration. Dotted lines: Standardized true values.

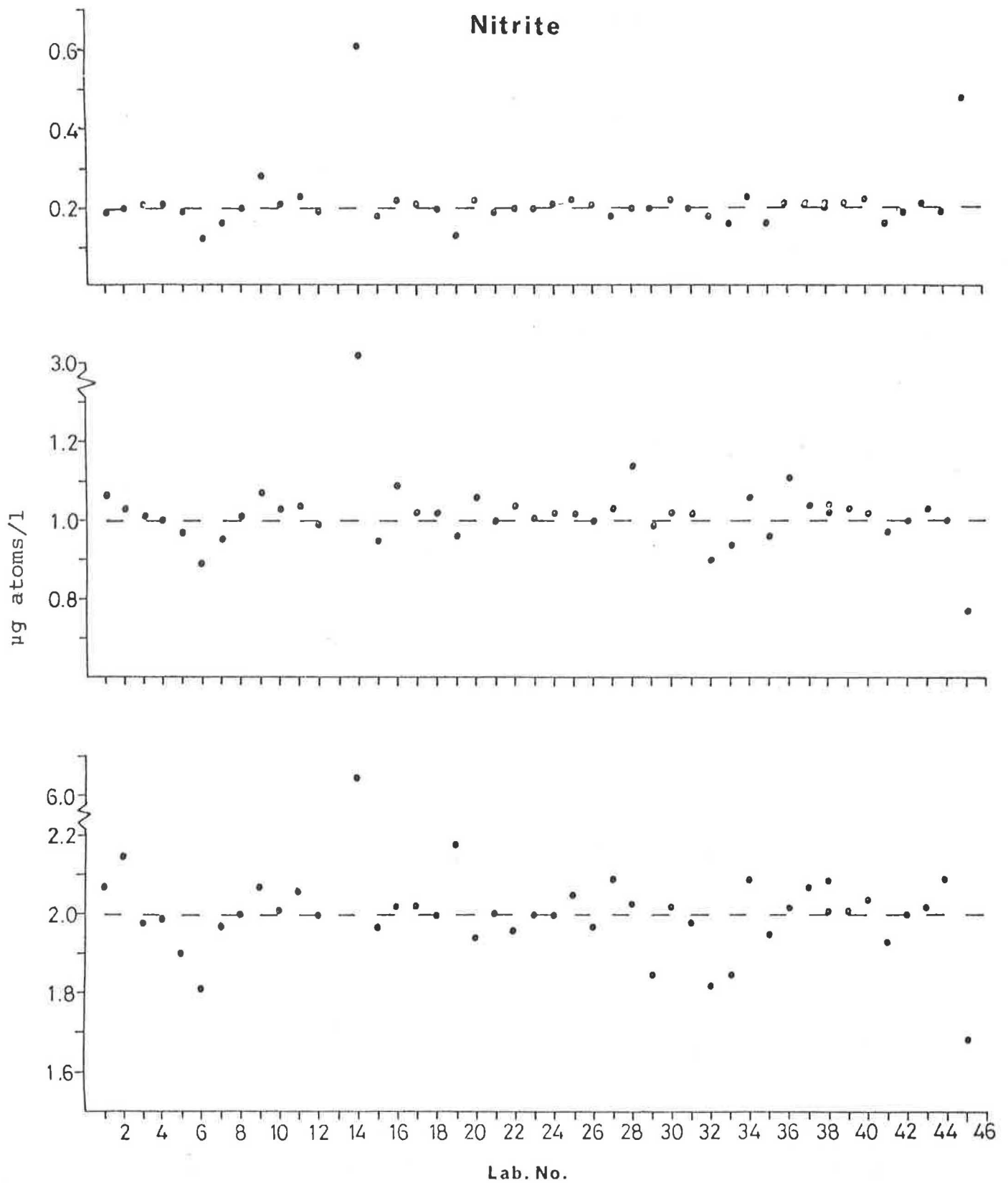


Fig.3. Standardized mean values of nitrite measurements reported by the different laboratories for the low, medium and high concentration. Dotted lines: Standardized true values.

Silicate

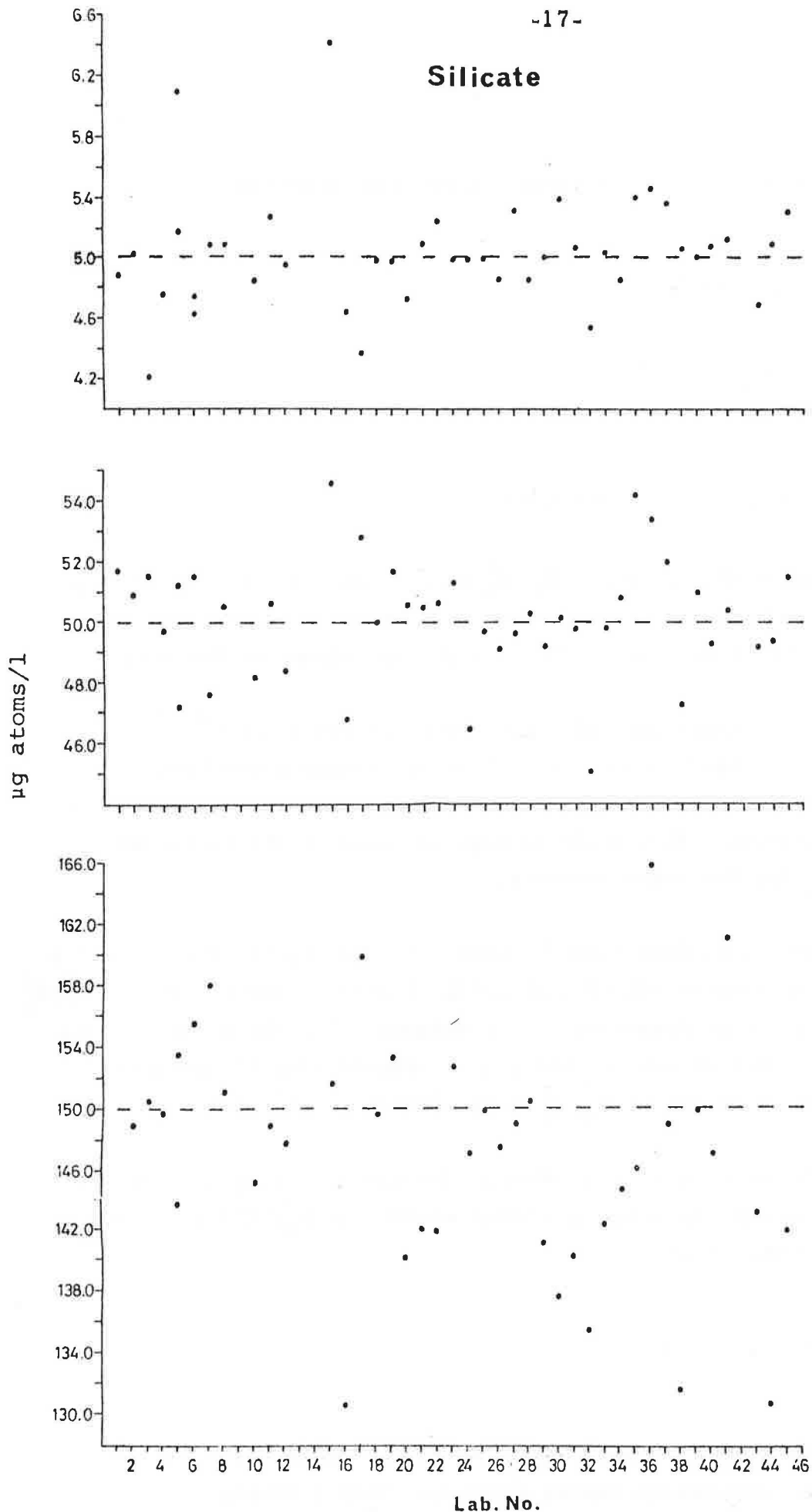


Fig.4. Standardized mean values of silicate measurements , reported by the different laboratories for the low, medium and high concentration. Dotted lines: Standardized true values.

where: $x_{i,j}$ = the j'th measurement in the i'th laboratory.

$$\bar{x}_i = \sum_{j=1}^3 \frac{x_{i,j}}{3}$$

$$\bar{x} = \sum_{i=1}^n \frac{\bar{x}_i}{n}$$

n = number of laboratories

By the method outlined above, S_w^2/S_b^2 was estimated for the different parameters and concentrations, both by splitting on method and by taking all methods together. The results are shown in Table 1.

It is difficult to detect possible differences in the ratio S_w^2/S_b^2 between methods used, since most of the laboratories used the same method and therefore only a few observations were made for each of the other methods. This leads to high variance in the estimates of the S_w^2/S_b^2 for the other methods.

When splitting on method Table 1. shows that for $n > 10$, the estimates of S_w^2/S_b^2 vary between 19.9% and 2.5%. For $n < 10$, the variation in S_w^2/S_b^2 is larger, but by disregarding a few outlying data, the estimates are of the same order of size as for $n > 10$. Considering all methods together, the estimates of S_w^2/S_b^2 vary between 31.7% and 2.1%.

These results show that the portion in the variance arising from the "within laboratory" variance is rather small. If $S_w^2/S_b^2 = 0.3$, then equation (1) shows that

$$\frac{S_w^2/3}{S^2} = 0.09$$

i.e., only 9% of the variance on a laboratory mean value on 3 replicate measurements comes from the "within laboratory" component of the variance.

For practical purposes, it was therefore decided to assume in the further analysis that all laboratory mean values for each parameter, concentration and method came from the same distribution, whatever the number of replicate measurements was.

Table 1 . Estimates of the ratio between "Within laboratory variance" (S_w^2) and "Between laboratory variance" (S_b^2). In brackets Numbers of laboratories.

Parameter	Method	S_w^2/S_b^2		
		low concentr.	med. concentr.	high concentr.
Phosphate	M	0.199(24)	0.0292(23)	0.1056(24)
	C	0.039(9)	0.0505(8)	0.098 (9)
	S	($S_b^2 < 0.0$) (3)	0.161 (3)	0.259 (3)
	All	0.147(36)	0.041 (34)	0.138 (36)
Nitrate	G	0.255(3)	0.012 (3)	0.030 (3)
	U	0.163(18)	0.119 (19)	0.034 (18)
	H	0.784(5)	0.760 (4)	0.141 (4)
	Z	0.036(2)	0.048 (2)	0.027 (2)
	All	0.226(29)	0.072 (29)	0.035 (28)
Nitrite		0.168(35)	0.125 (35)	0.021 (35)
Silicate	Y	8.11 (7)	0.269 (8)	0.096 (7)
	B	0.153(32)	0.025 (29)	0.041 (27)
	All	0.317(39)	0.062 (37)	0.043 (34)

(2) Precision and Accuracy for Different Parameters and Methods

Differences in variance between different methods were tested using the F-test at a 5% significance level. Average deviations from true value were tested using a t-test at a 5% significance level.

a. Phosphate (Table 2). Coefficient of variations for all methods together varied from 4.2% (high concentration) to 15.6% (low concentration). No significant differences in variance between methods were found. For method M, the average deviation from true value was significantly below zero for the low concentration, while, for method C, the average deviations were significantly below zero for the medium and high concentration. Considering all methods together, the average deviation from true value was significantly below zero for the low and high concentration.

b. Nitrate (Table 3). Coefficient for variation of all methods together varied from 9.4% (high concentration) to 20.6% (low concentration). The variance for method U was significantly lower than the variance for method G for all three levels of concentration. The method U gave, however, an average value significantly below the true value, both for the medium and the high concentration. Method H seemed to give too high values, significant at 5% level for medium concentration and significant at a 10% level for high concentration.

c. Nitrite (Table 4). Coefficient of variation varied from 4.1% (high concentration) to 12.0% (low concentration). The average value was higher than the true value for all three levels of concentration, but the difference was significant only for the medium concentration.

d. Silicate (Table 5). Coefficient of variation varied from 4.1% (medium concentration) to 7.2% (low concentration) for both methods combined. Variance for method B was higher than the variance for method Y for all concentrations, but the difference was significant only for the high concentration. The use of the same cell size for the measurement of the high concentration as used for medium and low concentration might account for the high variance for method B. The average value for method B was significantly lower than the true value for the high concentration.

Table 2. Variance of the phosphate measurements

Concentration	Method	Number	St. Dev.	CV(%)	Ave. dev. from true value.
Low, standardized value: 0.25 µg at/l	M	29	0.0327	14.23	-0.0203 ▼
	C	9	0.0507	22.24	-0.0220
	S	5	0.0228	8.95	0.0048
	all methods	43	0.0363	15.64	-0.0177 ▼
Medium, standardized value: 1.0 µg at/l	M	28	0.0787	8.01	-0.0175
	C	9	0.0767	8.23	-0.0678 ▼
	S	5	0.0756	7.31	0.0350
	all methods	42	0.0816	8.34	-0.0220
High, standardized value: 3.0 µg at/l.	M	29	0.1173	3.95	-0.0276
	C	9	0.1297	4.48	-0.1053 ▼
	S	5	0.1347	4.57	-0.0533
	all methods	43	0.1229	4.16	-0.0468 ▼

▼ Significant different from 0 (5% level of significance).

Table 3. Variance of the nitrate measurements

Concentration	Method	Number	St. Dev.	CV(%)	Ave. dev. from true value.
Low, standardized value: 0.5 µg at/l	C	9	0.1575	29.86	0.0276
	U	25	0.0773	15.64	-0.0057
	H	5	0.0835	16.46	0.0073
	T	1	-	-	-0.2222
	Z	2	0.0578	12.12	-0.0227
	all methods	42	0.1025	20.63	-0.0030
Medium, standardized value: 10.0 µg at/l	G	9	1.5223	15.40	-0.0079
	U	25	0.6675	6.87	-0.2892 ▼
	H	5	0.8117	7.15	1.3389 ▼
	T	1	-	-	-0.7778
	Z	2	0.5142	5.60	-0.8182
	all methods	42	1.0461	10.56	-0.0955
High, standardized value: 30.0 µg at /l	G	9	3.433	12.21	-1.902
	U	24	1.555	5.33	-0.8434 ▼
	H	5	3.472	10.37	3.468
	T	1	-	-	-0.1852
	Z	2	0.8142	2.96	-2.515
	all methods	41	2.764	9.40	-0.6155

▼ Significant different from 0 (5% level of significance)

Table 4. Variance of the nitrite measurements.

Concentration	Number	St.Dev.	CV(%)	Ave.dev.from true value
0.20 $\mu\text{g at/l}$ (low)	42	0.0240	12.02	0.0002
1.00 " (medium)	42	0.0448	4.41	0.0160 ▼
2.00 " (high)	42	0.0828	4.11	0.0148

▼ Significant different from 0 (5% level of significance)

Table 5. Variance of the silicate measurements.

Concentration	Method	Number	St.Dev.	CV(%)	Ave.dev.from true value.
Low, standardized value: 5.0 $\mu\text{g at/l}$	Y	7	0.2324	4.72	-0.0795
	B	36	0.3786	7.44	0.0902
	all methods	43	0.3622	7.15	0.0626
Medium, standardized value: 50.0 $\mu\text{g at/l}$	Y	8	1.802	3.64	-0.5068
	B	34	2.089	4.15	0.3848
	all methods	42	2.047	4.07	0.2150
High, standardized value: 150 $\mu\text{g at/l}$	Y	8	3.920	2.59	1.324
	B	34	8.232	5.63	-3.686 ▼
	all methods	42	7.819	5.30	-2.732

▼ Significant different from 0 (5% level of significance)

DISCUSSION

Inspection of Tables 2-5 shows that average deviations from the "true" value are negative to a significant degree for phosphate (-1.6%, -2.2% and -7.1% for the high, medium and low values), whereas for the other standards the average deviations from the true values are generally less than 2%. It appears, therefore, that only in the case of phosphate some deterioration of the standards may have occurred. Thus, with the possible exception of phosphate, the Sagami nutrient standards appear satisfactory.

The next most important observation is that only 9% of the variance on the laboratory values comes from the "within laboratory" component of the variance. Thus the precision of the various participating laboratories is much better than the overall accuracy. Inspection of the data in Table 7-10 clearly verifies that, even for laboratories with large deviations from the averages, precisions are generally satisfactory. This, therefore, suggests that the standardization procedures of the laboratories concerned are perhaps inadequate. Clearly these laboratories should reevaluate their procedures.

Tables 2-5 and Figures 1-4 make it abundantly clear that, as a whole, precisions for the methods for nitrite and silica are encouraging, though clearly some improvement in the determination of silica is warranted. Recent work on the Atlantic and Pacific Ocean has indicated that silica, if obtained with sufficiently high precision, can serve as a most useful oceanic tracer.

The situation for phosphate and nitrate is less satisfactory. This is particularly true for the latter, where the overall coefficients of variation are 21%, 11% and 9.5% at the low, medium, and high concentrations (Table 3). The method U (reduction in copper-treated cadmium column), used by the majority of the participants, gave data with a significantly smaller coefficient of variance, but with average values slightly below the overall average (about 2.5% at the medium and high levels). Notwithstanding this, it appears that this

method holds the most promise for precision, and clearly it is also the method preferred by the majority of the participants. The phosphate methods need much careful attention. The mixed reagents method seems preferable, but procedures should be refined to yield more consistently precise data.

CONCLUSIONS

In general, the intercalibration exercise proved to be most useful. Individual participants can use this compilation in efforts to make their data agree more with those obtained by other laboratories. As methods did not diverge much, it is clear that variations must be sought primarily in standardization procedures. The results will also aid participants in reevaluating their analytical procedures by comparison of their methods with those that appear most satisfactory from this exercise.

Uniform standards are of great importance in the analysis for nutrients. Two parallel paths can be suggested here:

- (1) Use of Sagami or equal quality standards; and
- (2) Use of standardized preparation procedures for primary standards.

Such procedures should leave no room for ambiguities to be introduced.

On the recommendation of the Working Group it was decided (C.Res. 1970/3:6): the nutrient salt standards prepared at the Sagami Chemical Research Center, for the exercise be the primary standard for all future ICES international and multi-ship expeditions. We also strongly recommend that, wherever this is hydrographically appropriate, a standard reference station be established in the study area.

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Finally, we would also take the opportunity to thank all the participating laboratories all over the world for the effort they have put into this intercalibration.

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from the highest to the lowest for each parameter.

[illegible][illegible][illegible][illegible][illegible][illegible]

Table 6 , continued. Standardized true and mean observed values ranged from the highest to the lowest for each parameter.

NITRITE		TRUE VALUE 0.200													
LAB.	14	45	9	34	11	16	25	20	30	40	37	3	36	38	
LAB.	43	17	24	38	4	39	26	10	18	29	23	31	2	22	
LAB.	8	28	21	5	1	12	44	42	15	32	27	7	33	41	
LAB.	35	19	6												
MEAN	0.611	0.478	0.278	0.233	0.230	0.222	0.221	0.219	0.216	0.215	0.212	0.212	0.211	0.211	
MEAN	0.211	0.211	0.211	0.211	0.210	0.207	0.206	0.206	0.203	0.202	0.200	0.200	0.200	0.200	
MEAN	0.200	0.197	0.193	0.190	0.189	0.188	0.188	0.185	0.182	0.182	0.181	0.164	0.161	0.159	
MEAN	0.158	0.133	0.119												

NITRITE		TRUE VALUE 1.000													
LAB.	14	28	36	16	9	34	20	1	38	11	37	22	39	43	
LAB.	30	10	27	2	25	17	24	38	18	31	40	3	8	23	
LAB.	21	42	44	4	26	12	29	41	5	35	19	7	15	33	
LAB.	32	6	45												
MEAN	3.222	1.138	1.111	1.093	1.067	1.061	1.056	1.056	1.044	1.044	1.038	1.035	1.034	1.033	
MEAN	1.029	1.028	1.026	1.026	1.023	1.022	1.022	1.022	1.020	1.017	1.014	1.011	1.011	1.011	
MEAN	1.007	1.003	0.999	0.997	0.996	0.993	0.987	0.967	0.964	0.963	0.957	0.954	0.945	0.939	
MEAN	0.908	0.893	0.772												

NITRITE		TRUE VALUE 2.000													
LAB.	14	36	16	19	34	38	27	44	37	9	1	11	2	25	
LAB.	40	39	10	38	28	43	30	17	8	12	42	23	21	24	
LAB.	18	4	31	3	26	7	15	22	35	20	41	5	29	33	
LAB.	32	6	45												
MEAN	6.500	2.237	2.191	2.175	2.094	2.094	2.093	2.090	2.069	2.067	2.067	2.056	2.048	2.045	
MEAN	2.041	2.037	2.036	2.033	2.029	2.022	2.021	2.019	2.008	2.005	2.003	2.000	2.000	2.000	
MEAN	1.996	1.989	1.981	1.977	1.969	1.966	1.966	1.957	1.951	1.937	1.930	1.904	1.852	1.851	
MEAN	1.817	1.811	1.676												

SILICATE		TRUE VALUE 5.000													
LAB.	15	5	36	35	30	37	27	45	11	22	3	5	41	21	
LAB.	7	8	44	40	31	38	33	39	2	29	25	24	23	18	
LAB.	19	12	1	34	26	28	10	4	20	6	43	16	6	32	
LAB.	17														
MEAN	6.424	6.091	5.462	5.412	5.395	5.373	5.322	5.296	5.270	5.248	5.209	5.194	5.133	5.097	
MEAN	5.091	5.091	5.091	5.082	5.073	5.055	5.036	5.026	5.022	5.000	5.000	4.989	4.989	4.976	
MEAN	4.970	4.939	4.878	4.852	4.848	4.848	4.842	4.745	4.734	4.734	4.693	4.636	4.619	4.536	
MEAN	4.368														

SILICATE		TRUE VALUE 50.000													
LAB.	15	35	41	36	17	37	1	17	6	3	45	23	5	37	
LAB.	2	34	22	20	11	8	21	28	30	18	33	31	4	25	
LAB.	27	44	40	43	29	26	12	10	7	38	5	16	24	32	
MEAN	54.641	54.159	53.883	53.392	52.802	51.959	51.696	51.656	51.549	51.530	51.463	51.327	51.175	50.959	
MEAN	50.685	50.811	50.633	50.619	50.601	50.542	50.470	50.332	50.133	50.030	49.822	49.759	49.705	49.699	
MEAN	49.558	49.367	49.313	49.226	49.207	49.126	48.373	48.180	47.559	47.308	47.167	46.836	46.460	45.118	

SILICATE		TRUE VALUE 150.000													
LAB.	36	41	17	1	7		5	19	23	15	8		3	39	
LAB.	25	4	18	27	11	2	37	12	26	24	40	35	10	34	
LAB.	5	43	33	21	45	22	29	31	20	30	32	38	44	16	
MEAN	165.929	161.136	159.682	158.075	156.022	155.531	153.528	153.257	152.729	151.628	151.086	150.483	150.461	149.926	
MEAN	149.879	149.742	149.578	148.968	148.894	148.894	148.884	147.768	147.467	147.124	147.105	146.110	145.205	144.764	
MEAN	143.577	143.215	142.400	142.099	142.085	142.039	141.194	140.350	140.177	137.684	135.525	131.637	130.730	130.494	

Table 7. Phosphate package A, lab.code, method used, true value in $\mu\text{g at/dm}^3$, number of replicates, mean value, variance and observed values.

PHOSPHATE		A							
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES			
7	M	0.275	3	0.1433	0.0000333	0.140	0.140	0.150	
7	M	1.101	3	0.7833	0.0016333	0.830	0.760	0.760	
7	M	3.164	3	3.5433	0.0044333	3.470	3.600	3.560	
8	M	0.275	3	0.2700	0.0000000	0.270	0.270	0.270	
8	M	1.101	3	1.0900	0.0000000	1.090	1.090	1.090	
8	M	3.164	3	3.1433	0.0004334	3.120	3.160	3.150	
19	M	0.275	3	0.2443	0.0000333	0.251	0.241	0.241	
19	M	1.101	3	1.0730	0.0000000	1.073	1.073	1.073	
19	M	3.164	3	3.1357	0.0000584	3.134	3.144	3.129	
21	M	0.275	4	0.2575	0.0000250	0.260	0.250	0.260	0.260
21	M	1.101	4	1.0550	0.0000333	1.050	1.050	1.060	1.060
21	M	3.164	4	3.0525	0.0000917	3.050	3.060	3.060	3.040
25	M	0.275	3	0.2667	0.0000333	0.260	0.270	0.270	
25	M	1.101	3	0.9933	0.0000333	0.990	0.990	1.000	
25	M	3.164	3	3.1867	0.0002334	3.200	3.170	3.190	
29	M	0.275	3	0.2600	0.0000000	0.260	0.260	0.260	
29	M	1.101	3	1.1100	0.0000000	1.110	1.110	1.110	
29	M	3.164	3	3.1200	0.0000000	3.120	3.120	3.120	
31	M	0.275	3	0.2567	0.0000333	0.260	0.250	0.260	
31	M	1.101	3	1.0333	0.0000333	1.030	1.030	1.040	
31	M	3.164	3	2.9800	0.0003000	2.990	2.990	2.960	
42	M	0.275	1	0.2500	0.0000000	0.250			
42	M	1.101	5	1.1460	0.0003300	1.120	1.150	1.140	1.170 1.150
42	M	3.164	5	3.4340	0.0000800	3.425	3.445	3.435	3.440 3.425
28	S	0.275	3	0.2967	0.0002333	0.310	0.280	0.300	
28	S	1.101	3	1.1000	0.0003000	1.090	1.120	1.090	
28	S	3.164	3	3.1667	0.0002333	3.180	3.170	3.150	
18	A C	0.275	3	0.2300	0.0000000	0.230	0.230	0.230	
18	A C	1.101	3	1.0267	0.0005333	1.000	1.040	1.040	
18	A C	3.164	3	2.9467	0.0142333	2.810	3.030	3.000	
46	A M	0.275	1	0.2100	0.0000000	0.210			
46	A M	1.101	4	1.0425	0.0117583	0.920	1.180	1.060	1.010
46	A M	3.164	5	2.9960	0.0205300	3.250	2.900	2.940	2.940 2.950

Table 7, continued. Phosphate package AC.

PHOSPHATE			AC					
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES		
10	M	0. 275	3	0. 2730	0. 0000120	0. 277	0. 271	0. 271
10	M	0. 901	3	0. 8980	0. 0001560	0. 908	0. 902	0. 884
10	M	2. 861	3	2. 8520	0. 0001560	2. 866	2. 842	2. 848
12	M	0. 275	3	0. 2533	0. 0002333	0. 240	0. 270	0. 250
12	M	0. 901	3	0. 8633	0. 0004333	0. 880	0. 870	0. 840
12	M	2. 861	3	2. 7667	0. 0001333	2. 780	2. 760	2. 760
22	M	0. 275	3	0. 2500	0. 0000000	0. 250	0. 250	0. 250
22	M	0. 901	3	0. 8600	0. 0000000	0. 860	0. 860	0. 860
22	M	2. 861	3	2. 7400	0. 0000000	2. 740	2. 740	2. 740
26	M	0. 275	3	0. 2900	0. 0000000	0. 290	0. 290	0. 290
26	M	0. 901	3	0. 9367	0. 0001333	0. 930	0. 930	0. 950
26	M	2. 861	3	2. 8233	0. 0000334	2. 820	2. 820	2. 830
32	M	0. 275	3	0. 2600	0. 0000000	0. 260	0. 260	0. 260
32	M	0. 901	3	1. 0500	0. 0000000	1. 050	1. 050	1. 050
32	M	2. 861	3	3. 0100	0. 0000000	3. 010	3. 010	3. 010
33	M	0. 275	3	0. 2807	0. 0000083	0. 279	0. 279	0. 284
33	M	0. 901	3	1. 0123	0. 0000013	1. 013	1. 013	1. 011
33	M	2. 861	3	3. 0000	0. 0000000	3. 000	3. 000	3. 000
37	M	0. 275	3	0. 2633	0. 0021333	0. 210	0. 290	0. 290
37	M	0. 901	3	0. 7800	0. 0001000	0. 780	0. 770	0. 790
37	M	2. 861	3	2. 7967	0. 0097334	2. 910	2. 730	2. 750
40	M	0. 275	3	0. 1400	0. 0000000	0. 140	0. 140	0. 140
40	M	0. 901	3	0. 8233	0. 0001333	0. 810	0. 830	0. 830
40	M	2. 861	3	2. 8433	0. 0008334	2. 810	2. 860	2. 860
44	M	0. 275	3	0. 2100	0. 0000000	0. 210	0. 210	0. 210
44	M	0. 901	3	0. 8367	0. 0001333	0. 830	0. 830	0. 850
44	M	2. 861	3	2. 8667	0. 0033334	2. 800	2. 900	2. 900
5	C	0. 275	3	0. 2800	0. 0003000	0. 290	0. 260	0. 290
5	C	0. 901	4	0. 8775	0. 0002250	0. 870	0. 870	0. 870 0. 900
5	C	2. 861	3	2. 8400	0. 0000000	2. 840	2. 840	2. 840
15	C	0. 275	3	0. 3700	0. 0000000	0. 370	0. 370	0. 370
15	C	0. 901	3	0. 8800	0. 0000000	0. 880	0. 880	0. 880
15	C	2. 861	3	2. 8567	0. 0001333	2. 850	2. 870	2. 850
35	S	0. 275	3	0. 2800	0. 0016000	0. 240	0. 320	0. 280
35	S	0. 901	3	0. 8600	0. 0016000	0. 820	0. 900	0. 860
35	S	2. 861	3	2. 6100	0. 0111000	2. 500	2. 710	2. 620

Table 7, continued. Phosphate package B + BC.

PHOSPHATE			B+BC					
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES		
2	M	0.225	3	0.1933	0.0001333	0.200	0.200	0.180
2	M	0.901	3	0.8167	0.0001333	0.810	0.810	0.830
2	M	2.861	3	2.8033	0.0000333	2.800	2.810	2.800
3	M	0.225	3	0.1800	0.0001000	0.190	0.170	0.180
3	M	2.861	3	2.7367	0.0021334	2.710	2.710	2.790
9	M	0.225	3	0.2200	0.0001000	0.230	0.220	0.210
9	M	0.901	3	0.9000	0.0007000	0.890	0.930	0.880
9	M	2.861	3	2.9133	0.0001334	2.920	2.900	2.920
11	M	0.225	3	0.2233	0.0001333	0.210	0.230	0.230
11	M	0.901	3	0.9467	0.0010333	0.970	0.960	0.910
11	M	2.861	3	2.9000	0.0007000	2.910	2.920	2.870
14	M	0.225	2	0.3300	0.0050000	0.280	0.380	
14	M	0.901	3	1.5000	0.0000000	1.500	1.500	1.500
14	M	2.861	3	5.0667	0.0033334	5.000	5.100	5.100
16	M	0.225	3	0.2200	0.0000000	0.220	0.220	0.220
16	M	0.901	3	0.9200	0.0003000	0.900	0.930	0.930
16	M	2.861	3	2.6367	0.0021333	2.610	2.690	2.610
23	M	0.225	3	0.2017	0.0000083	0.205	0.200	0.200
23	M	0.901	3	0.8780	0.0000270	0.875	0.875	0.884
23	M	2.861	3	2.8520	0.0000270	2.858	2.849	2.849
24	M	0.225	3	0.2233	0.0001333	0.230	0.230	0.210
24	M	0.901	3	0.9200	0.0000000	0.920	0.920	0.920
24	M	2.861	3	2.9000	0.0000000	2.900	2.900	2.900
27	M	0.225	3	0.2100	0.0004000	0.190	0.210	0.230
27	M	0.901	3	0.8600	0.0001000	0.850	0.860	0.870
27	M	2.861	3	2.7267	0.0002334	2.710	2.730	2.740
34	M	0.225	2	0.2945	0.0004205	0.280	0.309	
34	M	0.901	2	0.9925	0.0006125	1.010	0.975	
34	M	2.861	2	3.0700	0.0008000	3.090	3.050	
39	M	0.225	3	0.2000	0.0000000	0.200	0.200	0.200
39	M	0.901	3	0.8733	0.0000333	0.870	0.880	0.870
39	M	2.861	3	2.8200	0.0001000	2.830	2.820	2.810

Table 7, continued. Phosphate package B + BC.

PHOSPHATE			B+BC							
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES				
45	M	0.225	1	0.1700	0.0000000	0.170				
45	M	0.901	5	0.9280	0.0023700	0.910	1.010	0.920	0.920	0.880
45	M	2.861	5	2.8300	0.0056000	2.820	2.760	2.760	2.880	2.930
1	C	0.225	3	0.2067	0.0001333	0.200	0.220	0.200		
1	C	0.901	3	0.8633	0.0000333	0.860	0.860	0.870		
1	C	2.861	3	2.8533	0.0000334	2.850	2.860	2.850		
4	C	0.225	3	0.2100	0.0000000	0.210	0.210	0.210		
4	C	0.901	3	0.8417	0.0000003	0.842	0.841	0.842		
4	C	2.861	3	2.7043	0.0000004	2.704	2.704	2.705		
6	C	0.225	3	0.2033	0.0000333	0.210	0.200	0.200		
6	C	0.901	3	0.8600	0.0004000	0.860	0.840	0.880		
6	C	2.861	3	2.8033	0.0000333	2.800	2.800	2.810		
17	C	0.225	3	0.1933	0.0000333	0.190	0.190	0.200		
17	C	0.901	3	0.9267	0.0000333	0.920	0.930	0.930		
17	C	2.861	3	2.8133	0.0001333	2.800	2.820	2.820		
38	C	0.225	3	0.1300	0.0003000	0.120	0.120	0.150		
38	C	0.901	3	0.7833	0.0012333	0.750	0.780	0.820		
38	C	2.861	3	2.8267	0.0005334	2.840	2.840	2.800		
30	S	0.225	1	0.1980	0.0000000	0.198				
30	S	0.901	1	1.0230	0.0000000	1.023				
30	S	2.861	1	2.8840	0.0000000	2.884				
36	S	0.225	3	0.2520	0.0000640	0.244	0.260	0.252		
36	S	0.901	3	0.9843	0.0000563	0.977	0.992	0.984		
36	S	2.861	3	2.9333	0.0094103	2.837	3.031	2.932		
43	S	0.225	2	0.2250	0.0000500	0.230	0.220			
43	S	0.901	2	0.8950	0.0000500	0.900	0.890			
43	S	2.861	2	2.7600	0.0000000	2.760	2.760			
20	A M	0.225	3	0.1967	0.0001333	0.190	0.190	0.210		
20	A M	0.901	3	0.7000	0.0001000	0.690	0.710	0.700		
20	A M	2.861	3	2.6700	0.0004000	2.650	2.670	2.690		
41	A C	0.225	3	0.1833	0.0000333	0.180	0.180	0.190		
41	A C	0.901	3	0.6867	0.0000333	0.690	0.690	0.680		
41	A C	2.861	3	2.4833	0.0005333	2.470	2.470	2.510		

Table 8. Nitrate package A + AC, lab.code, method used, true value in $\mu\text{g at/dm}^3$, number of replicates, mean value, variance and observed values.

NITRATE			A+AC					
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES		
12	G	0.550	3	0.8000	0.0100000	0.700	0.800	0.900
12	G	11.000	3	12.3667	0.0133336	12.300	12.300	12.500
12	G	33.000	3	34.9333	0.0233345	34.800	34.900	35.100
15	G	0.550	3	0.6033	0.0000333	0.600	0.610	0.600
15	G	11.000	3	12.2933	0.0016333	12.340	12.270	12.270
15	G	33.000	3	33.2833	0.0085354	33.390	33.230	33.230
33	G	0.550	2	0.9100	0.0018000	0.880	0.940	
33	G	11.000	2	12.8500	0.0050000	12.800	12.900	
33	G	33.000	2	32.7000	0.0000019	32.700	32.700	
37	G	0.550	2	0.5800	0.0000000	0.580	0.580	
37	G	11.000	2	10.8500	0.2450002	10.500	11.200	
37	G	33.000	2	30.8500	0.0050006	30.900	30.800	
42	G	0.550	1	0.3970	0.0000000	0.397		
42	G	11.000	1	11.0600	0.0000000	11.060		
42	G	33.000	1	31.9200	0.0000000	31.920		
8	U	0.550	2	0.6000	0.0000000	0.600	0.600	
8	U	11.000	2	10.9000	0.0000001	10.900	10.900	
8	U	33.000	2	31.8000	0.0199995	31.700	31.900	
10	U	0.550	3	0.5883	0.0000203	0.584	0.593	0.588
10	U	11.000	3	11.1100	0.0144001	11.230	10.990	11.110
10	U	33.000	3	33.3833	0.0770340	33.680	33.340	33.130
19	U	0.550	3	0.5767	0.0006333	0.580	0.600	0.550
19	U	11.000	3	11.4100	0.0084002	11.330	11.390	11.510
19	U	33.000	3	31.9367	0.0400338	31.740	32.140	31.930
22	U	0.550	2	0.5050	0.0004500	0.490	0.520	
22	U	11.000	2	10.9150	0.0220500	10.810	11.020	
22	U	33.000	2	32.0950	0.2520494	32.450	31.740	
26	U	0.550	3	0.6000	0.0000000	0.600	0.600	0.600
26	U	11.000	3	11.4000	0.0100002	11.300	11.500	11.400
26	U	33.000	3	33.2667	0.0033350	33.200	33.300	33.300

Table 8, continued. Nitrate package A + AC.

NITRATE			A+AC						
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES			
28	U	0.550	3	0.4400	0.0001000	0.440	0.430	0.450	
28	U	11.000	3	10.2300	0.0019001	10.210	10.200	10.280	
28	U	33.000	3	29.9967	0.0000353	30.000	29.990	30.000	
29	U	0.550	3	0.5400	0.0000000	0.540	0.540	0.540	
29	U	11.000	3	11.1600	0.0000002	11.160	11.160	11.160	
29	U	33.000	3	31.7600	0.0000005	31.760	31.760	31.760	
31	U	0.550	3	0.5667	0.0005333	0.540	0.580	0.580	
31	U	11.000	3	10.6000	0.0400001	10.400	10.800	10.600	
31	U	33.000	3	28.8333	1.0133352	28.700	29.900	27.900	
35	U	0.550	2	0.6950	0.0012500	0.720	0.670		
35	U	11.000	2	9.7900	0.0098001	9.860	9.720		
40	U	0.550	3	0.5067	0.0000333	0.500	0.510	0.510	
40	U	11.000	3	8.8100	0.0007000	8.780	8.820	8.830	
40	U	33.000	3	27.6800	0.0012021	27.660	27.660	27.720	
44	U	0.550	3	0.5133	0.0017333	0.500	0.560	0.480	
44	U	11.000	3	10.7000	0.0000000	10.700	10.700	10.700	
44	U	33.000	3	31.5000	0.0000000	31.500	31.500	31.500	
18	A U	0.550	3	0.3900	0.0001000	0.380	0.390	0.400	
18	A U	11.000	3	11.0667	0.0133334	11.200	11.000	11.000	
18	A U	33.000	3	32.0000	0.0400028	32.200	32.000	31.800	
5	H	0.550	3	0.4300	0.0000000	0.430	0.430	0.430	
5	H	11.000	4	11.2300	0.2658002	11.640	11.070	10.570	11.640
5	H	33.000	6	32.2000	2.5360008	32.700	32.400	33.600	30.700 29.900 33.900
25	Z	0.550	3	0.5700	0.0003000	0.590	0.560	0.560	
25	Z	11.000	3	10.5000	0.0300000	10.300	10.600	10.600	
25	Z	33.000	3	30.8667	0.0133352	31.000	30.800	30.800	
32	Z	0.550	3	0.4800	0.0000000	0.480	0.480	0.480	
32	Z	11.000	3	9.7000	0.0000000	9.700	9.700	9.700	
32	Z	33.000	3	29.6000	0.0300007	29.500	29.500	29.800	

Table 8, continued. Nitrate package B + BC.

NITRATE			B+BC					
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES		
2	G	0.450	2	0.3750	0.0084500	0.310	0.440	
2	G	9.000	2	9.1500	0.1682001	8.860	9.440	
2	G	27.000	2	26.5500	0.8449998	25.900	27.200	
4	G	0.450	3	0.4717	0.0003083	0.455	0.470	0.490
4	G	9.000	3	8.7933	0.0096333	8.850	8.680	8.850
4	G	27.000	3	26.8333	0.0533347	26.700	26.700	27.100
14	G	0.450	2	0.6500	0.0450000	0.500	0.800	
14	G	9.000	2	6.3500	0.4050001	5.900	6.800	
14	G	27.000	2	17.9000	2.4200006	16.800	19.000	
16	G	0.450	2	0.3650	0.0012500	0.340	0.390	
16	G	9.000	2	7.0450	0.0264501	6.930	7.160	
16	G	27.000	2	21.1350	0.0264506	21.020	21.250	
45	G	0.450	1	0.3700	0.0000000	0.370		
45	G	9.000	1	6.4400	0.0000000	6.440		
45	G	27.000	1	19.1500	0.0000000	19.150		
1	U	0.450	3	0.2567	0.0000333	0.250	0.260	0.260
1	U	9.000	3	8.0667	0.1508334	7.750	8.500	7.950
1	U	27.000	3	23.5833	0.0033331	23.650	23.550	23.550
6	U	0.450	3	0.4467	0.0096333	0.560	0.390	0.390
6	U	9.000	3	7.4367	0.6165335	7.890	7.890	6.530
6	U	27.000	3	27.1100	0.2989011	26.480	27.390	27.460
9	U	0.450	3	0.4200	0.0003000	0.410	0.440	0.410
9	U	9.000	3	8.9633	0.0120335	9.050	9.000	8.840
9	U	27.000	3	27.2267	0.0377340	27.060	27.440	27.180
17	U	0.450	3	0.5133	0.0000333	0.510	0.510	0.520
17	U	9.000	3	9.2300	0.0000001	9.230	9.230	9.230
17	U	27.000	3	27.3333	0.0133352	27.400	27.400	27.200
21	U	0.450	3	0.4400	0.0000000	0.440	0.440	0.440
21	U	9.000	3	8.9300	0.0027001	8.960	8.960	8.870
21	U	27.000	3	26.8500	0.0236998	26.890	26.980	26.680
23	U	9.000	1	9.4900	0.0000000	9.490		
23	U	27.000	2	27.9500	0.0050015	27.900	28.000	

Table 8, continued. Nitrate package B + BC.

NITRATE			B+BC								
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES					
24	U	0.450	2	0.4700	0.0002000	0.480	0.500				
24	U	9.000	3	9.0000	0.0100000	9.100	8.900	9.000			
23	U	0.450	1	0.4700	0.0000000	0.470					
24	U	27.000	3	27.5000	0.0400000	27.700	27.300	27.500			
27	U	0.450	3	0.5467	0.0006333	0.520	0.550	0.570			
27	U	9.000	3	9.4800	0.0049000	9.430	9.450	9.560			
27	U	27.000	3	27.7000	0.0300012	27.500	27.800	27.800			
34	U	0.450	2	0.5550	0.0012500	0.530	0.580				
34	U	9.000	2	14.8000	0.0800000	15.000	14.600				
34	U	27.000	2	42.9500	0.0450029	42.800	43.100				
38	U	0.450	1	0.4500	0.0000000	0.450					
38	U	9.000	1	8.2200	0.0000000	8.220					
38	U	27.000	1	26.4800	0.0000000	26.480					
39	U	0.450	3	0.4867	0.0014333	0.530	0.470	0.460			
39	U	9.000	3	8.9333	0.0133333	8.800	9.000	9.000			
39	U	27.000	3	25.6333	0.1633334	25.200	26.000	25.700			
20	A U	0.450	3	0.3567	0.0000333	0.350	0.360	0.360			
20	A U	9.000	3	9.0967	0.0025334	9.150	9.050	9.090			
20	A U	27.000	6	26.9383	0.0476181	27.150	27.280	26.800	26.800	26.800	
38	A U	0.450	2	0.5050	0.0000500	0.510	0.500				
38	A U	9.000	2	8.7450	0.0112500	8.820	8.670				
38	A U	27.000	2	27.6000	0.0032001	27.640	27.560				
41	A U	0.450	3	0.4033	0.0001333	0.410	0.410	0.390			
41	A U	9.000	3	8.1000	0.0009001	8.100	8.130	8.070			
41	A U	27.000	3	26.2167	0.0166335	26.360	26.180	26.110			
3	H	0.450	3	0.5633	0.0021333	0.590	0.590	0.510			
3	H	9.000	3	10.9667	0.2133336	10.700	10.700	11.500			
3	H	27.000	3	33.4333	0.0033331	33.400	33.400	33.500			
11	H	0.450	3	0.4533	0.0012333	0.420	0.450	0.490			
11	H	9.000	3	9.7233	0.0908334	9.690	10.040	9.440			
11	H	27.000	3	27.2600	0.5092006	27.600	27.740	26.440			
30	H	0.450	3	0.4677	0.0135723	0.584	0.351	0.468			
30	H	9.000	3	10.4800	0.3738999	10.910	9.780	10.750			
30	H	27.000	3	32.0333	3.2933359	34.100	30.700	31.300			
36	H	0.450	3	0.4467	0.0006333	0.420	0.470	0.450			
36	H	9.000	3	10.6667	0.0033334	10.600	10.700	10.700			
36	H	27.000	3	31.5333	0.0033336	31.600	31.500	31.500			
43	T	0.450	3	0.2500	0.0049000	0.180	0.250	0.320			
43	T	9.000	3	8.3000	0.0100001	8.200	8.300	8.400			
43	T	27.000	3	26.8333	0.2033324	26.400	26.800	27.300			

Table 9. Nitrite package A + AC, lab. code, methode used, true value in $\mu\text{g at/dm}^3$, number of replicates, mean value, variance and observed values.

NITRITE			A+AC		VARIANCE	OBSERVED VALUES		
LAB	MET	T. VAL	N	MEAN				
5		0.220	3	0.2090	0.0000000	0.209	0.209	0.209
5		1.101	3	1.0613	0.0000653	1.052	1.066	1.066
5		2.201	3	2.0950	0.0000000	2.095	2.095	2.095
7		0.220	3	0.1800	0.0000000	0.180	0.180	0.180
7		1.101	3	1.0500	0.0000000	1.050	1.050	1.050
7		2.201	3	2.1633	0.0001333	2.170	2.170	2.150
8		0.220	3	0.2200	0.0000000	0.220	0.220	0.220
8		1.101	3	1.1133	0.0000333	1.110	1.110	1.120
8		2.201	3	2.2100	0.0001000	2.210	2.200	2.220
10		0.220	3	0.2263	0.0000013	0.227	0.227	0.225
10		1.101	3	1.1313	0.0000013	1.130	1.132	1.132
10		2.201	3	2.2403	0.0000053	2.239	2.239	2.243
12		0.220	3	0.2067	0.0005333	0.180	0.220	0.220
12		1.101	3	1.0933	0.0005333	1.080	1.120	1.080
12		2.201	3	2.2067	0.0005333	2.180	2.220	2.220
15		0.220	3	0.2000	0.0000000	0.200	0.200	0.200
15		1.101	3	1.0400	0.0000000	1.040	1.040	1.040
15		2.201	3	2.1633	0.0000333	2.170	2.160	2.160
19		0.220	3	0.1463	0.0000013	0.147	0.147	0.145
19		1.101	3	1.0533	0.0021763	1.049	1.009	1.102
19		2.201	3	2.3933	0.0001563	2.406	2.393	2.381
22		0.220	3	0.2200	0.0000000	0.220	0.220	0.220
22		1.101	3	1.1400	0.0000000	1.140	1.140	1.140
22		2.201	3	2.1533	0.0001333	2.140	2.160	2.160
25		0.220	3	0.2433	0.0005333	0.270	0.230	0.230
25		1.101	3	1.1267	0.0005333	1.140	1.100	1.140
25		2.201	3	2.2500	0.0000000	2.250	2.250	2.250
26		0.220	3	0.2267	0.0000333	0.220	0.230	0.230
26		1.101	3	1.0967	0.0000333	1.090	1.100	1.100
26		2.201	3	2.1667	0.0002333	2.150	2.170	2.180
28		0.220	3	0.2167	0.0001333	0.230	0.210	0.210
28		1.101	3	1.2533	0.0046333	1.330	1.230	1.200
28		2.201	3	2.2333	0.0002333	2.230	2.220	2.250

Table 9, continued. Nitrite package A + AC.

NITRITE			A+AC					
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES		
29		0.220	3	0.2220	0.0000000	0.222	0.222	0.222
29		1.101	3	1.0870	0.0000000	1.087	1.087	1.087
29		2.201	3	2.0383	0.0000333	2.045	2.035	2.035
31		0.220	3	0.2200	0.0000000	0.220	0.220	0.220
31		1.101	3	1.1200	0.0001000	1.110	1.130	1.120
31		2.201	3	2.1800	0.0001000	2.170	2.180	2.190
32		0.220	3	0.2000	0.0000000	0.200	0.200	0.200
32		1.101	3	1.0000	0.0000000	1.000	1.000	1.000
32		2.201	3	2.0000	0.0000000	2.000	2.000	2.000
33		0.220	3	0.1767	0.0000333	0.180	0.180	0.170
33		1.101	3	1.0333	0.0000333	1.030	1.030	1.040
33		2.201	3	2.0367	0.0000333	2.040	2.030	2.040
35		0.220	3	0.1733	0.0016333	0.220	0.150	0.150
35		1.101	3	1.0600	0.0013000	1.100	1.030	1.050
35		2.201	3	2.1467	0.0008333	2.180	2.130	2.130
37		0.220	3	0.2333	0.0000333	0.230	0.230	0.240
37		1.101	3	1.1433	0.0001333	1.130	1.150	1.150
37		2.201	3	2.2767	0.0002333	2.260	2.280	2.290
40		0.220	3	0.2367	0.0004333	0.220	0.230	0.260
40		1.101	3	1.1167	0.0001333	1.110	1.110	1.130
40		2.201	3	2.2467	0.0000333	2.240	2.250	2.250
42		0.220	1	0.2040	0.0000000	0.204		
42		1.101	1	1.1040	0.0000000	1.104		
42		2.201	1	2.2040	0.0000000	2.204		
44		0.220	3	0.2067	0.0001333	0.220	0.200	0.200
44		1.101	3	1.1000	0.0000000	1.100	1.100	1.100
44		2.201	3	2.3000	0.0000000	2.300	2.300	2.300
18	A	0.220	3	0.2233	0.0001333	0.210	0.230	0.230
18	A	1.101	3	1.1233	0.0000333	1.120	1.120	1.130
18	A	2.201	3	2.1967	0.0000333	2.200	2.190	2.200

Table 9, continued. Nitrite package B + BC.

NITRITE			B+BC							
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES				
1		0.180	2	0.1700	0.0000000	0.170	0.170			
1		0.900	2	0.9500	0.0000000	0.950	0.950			
1		1.800	2	1.8600	0.0000000	1.860	1.860			
2		0.180	3	0.1800	0.0000000	0.180	0.180	0.180		
2		0.900	3	0.9233	0.0000333	0.920	0.920	0.930		
2		1.800	3	1.8433	0.0000333	1.840	1.840	1.850		
3		0.180	3	0.1907	0.0000093	0.188	0.190	0.194		
3		0.900	3	0.9103	0.0000583	0.912	0.917	0.902		
3		1.800	3	1.7793	0.0000403	1.772	1.783	1.783		
4		0.180	3	0.1890	0.0000000	0.189	0.189	0.189		
4		0.900	3	0.8977	0.0000163	0.900	0.893	0.900		
4		1.800	3	1.7897	0.0000003	1.789	1.790	1.790		
6		0.180	3	0.1067	0.0000333	0.110	0.110	0.100		
6		0.900	3	0.8033	0.0005333	0.830	0.790	0.790		
6		1.800	3	1.6300	0.0000000	1.630	1.630	1.630		
9		0.180	3	0.2500	0.0001000	0.250	0.260	0.240		
9		0.900	3	0.9600	0.0001000	0.970	0.960	0.950		
9		1.800	3	1.8600	0.0003000	1.880	1.850	1.850		
11		0.180	3	0.2067	0.0000333	0.200	0.210	0.210		
11		0.900	3	0.9400	0.0001000	0.930	0.940	0.950		
11		1.800	3	1.8500	0.0001000	1.850	1.840	1.860		
14		0.180	2	0.5500	0.0050000	0.600	0.500			
14		0.900	2	2.9000	0.0200000	2.800	3.000			
14		1.800	2	5.8500	0.0050001	5.900	5.800			
16		0.180	5	0.2000	0.0001000	0.190	0.200	0.190	0.210	0.210
16		0.900	5	0.9840	0.0000800	0.980	0.980	0.980	1.000	0.980
16		1.800	5	1.9720	0.0003200	1.960	1.960	1.960	2.000	1.980
17		0.180	3	0.1900	0.0000000	0.190	0.190	0.190		
17		0.900	3	0.9200	0.0000000	0.920	0.920	0.920		
17		1.800	3	1.8167	0.0000333	1.820	1.810	1.820		
21		0.180	3	0.1733	0.0000333	0.170	0.180	0.170		
21		0.900	3	0.9067	0.0001333	0.900	0.920	0.900		
21		1.800	3	1.8000	0.0000000	1.800	1.800	1.800		
23		0.180	1	0.1800	0.0000000	0.180				
23		0.900	1	0.9100	0.0000000	0.910				
23		1.800	1	1.8000	0.0000000	1.800				

Table 9, continued. Nitrite package B + BC.

NITRITE			B+BC					
LAB	MEI	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES		
24		0.180	3	0.1900	0.0000000	0.190	0.190	0.190
24		0.900	3	0.9200	0.0000000	0.920	0.920	0.920
24		1.800	3	1.8000	0.0000000	1.800	1.800	1.800
27		0.180	3	0.1633	0.0001333	0.150	0.170	0.170
27		0.900	3	0.9233	0.0000333	0.920	0.920	0.930
27		1.800	3	1.8833	0.0001333	1.870	1.890	1.890
30		0.180	3	0.1947	0.0000083	0.193	0.193	0.198
30		0.900	3	0.9260	0.0000270	0.923	0.923	0.932
30		1.800	3	1.8193	0.0000083	1.821	1.816	1.821
34		0.180	2	0.2100	0.0000000	0.210	0.210	
34		0.900	2	0.9550	0.0000500	0.950	0.960	
34		1.800	2	1.8850	0.0000500	1.890	1.880	
36		0.180	3	0.1900	0.0000000	0.190	0.190	0.190
36		0.900	3	1.0000	0.0000000	1.000	1.000	1.000
36		1.800	3	2.0133	0.0000333	2.010	2.010	2.020
38		0.180	2	0.1900	0.0000000	0.190	0.190	
38		0.900	2	0.9400	0.0000000	0.940	0.940	
38		1.800	2	1.8850	0.0000500	1.880	1.890	
39		0.180	3	0.1863	0.0000013	0.187	0.187	0.185
39		0.900	3	0.9307	0.0000213	0.928	0.928	0.936
39		1.800	3	1.8333	0.0000163	1.838	1.831	1.831
43		0.180	3	0.1900	0.0000000	0.190	0.190	0.190
43		0.900	3	0.9300	0.0000000	0.930	0.930	0.930
43		1.800	3	1.8200	0.0000000	1.820	1.820	1.820
45		0.180	1	0.4300	0.0000000	0.430		
45		0.900	1	0.6950	0.0000000	0.695		
45		1.800	1	1.5080	0.0000000	1.508		
20	A	0.180	3	0.1967	0.0000333	0.190	0.200	0.200
20	A	0.900	3	0.9500	0.0001000	0.940	0.950	0.960
20	A	1.800	3	1.7433	0.0000333	1.740	1.750	1.740
38	A	0.180	2	0.1900	0.0002000	0.200	0.180	
38	A	0.900	2	0.9200	0.0000000	0.920	0.920	
38	A	1.800	2	1.8300	0.0000000	1.830	1.830	
41	A	0.180	3	0.1433	0.0000333	0.140	0.140	0.150
41	A	0.900	3	0.8700	0.0000000	0.870	0.870	0.870
41	A	1.800	3	1.7367	0.0012333	1.700	1.740	1.770

Table 10. Silicate package A + AC, lab.code, method used, true value in $\mu\text{g at/dm}^3$, number of replicates, mean value, variance and observed values.

SILICATE			A+AC		VARIANCE	OBSERVED VALUES					
LAB	MET	T. VAL	N	MEAN							
5	B	5.500	3	5.7133	0.0085334	5.820	5.660	5.660			
5	B	55.300	3	52.1667	0.2533321	52.100	52.700	51.700			
5	B	165.800	6	158.7000	5.3240601	162.100	160.100	158.100	157.400	155.400	159.100
8	B	5.500	3	5.6000	0.0000000	5.600	5.600	5.600			
8	B	55.300	3	55.9000	0.0000019	55.900	55.900	55.900			
8	B	165.800	3	167.0000	0.4799957	166.600	166.600	167.800			
10	B	5.500	3	5.3267	0.0002334	5.340	5.310	5.330			
10	B	55.300	3	53.2867	0.0240326	53.290	53.130	53.440			
10	B	165.800	3	160.5000	0.2700043	160.800	160.800	159.900			
12	B	5.500	3	5.4333	0.0033334	5.500	5.400	5.400			
12	B	55.300	3	53.5000	0.0000000	53.500	53.500	53.500			
12	B	165.800	3	163.3333	0.3333588	163.000	164.000	163.000			
15	B	5.500	3	7.0667	0.0033334	7.100	7.100	7.000			
15	B	55.300	3	60.4333	0.0533314	60.300	60.300	60.700			
15	B	165.800	3	167.6000	0.2700348	167.900	167.900	167.000			
21	B	5.500	3	5.6067	0.0001334	5.600	5.620	5.600			
21	B	55.300	3	55.8200	0.0291023	55.870	55.630	55.960			
21	B	165.800	3	157.0667	0.5733643	157.600	157.400	156.200			
22	B	5.500	3	5.7733	0.0001333	5.780	5.780	5.760			
22	B	55.300	3	56.0000	0.0027065	55.940	56.030	56.030			
22	B	165.800	3	157.0000	0.0000000	157.000	157.000	157.000			
25	B	5.500	3	5.5000	0.0100001	5.500	5.400	5.600			
25	B	55.300	3	54.9667	0.0533352	54.700	55.100	55.100			
25	B	165.800	3	165.6667	0.3333282	165.000	166.000	166.000			
29	B	5.500	3	5.5000	0.0000000	5.500	5.500	5.500			
29	B	55.300	3	54.4233	0.2670307	55.020	54.120	54.130			
29	B	165.800	3	156.0667	0.3333588	155.400	156.400	156.400			
31	B	5.500	6	5.5800	0.0047200	5.560	5.530	5.530	5.530	5.690	5.640
31	B	55.300	6	55.0333	0.2306702	54.200	54.800	55.200	55.200	55.200	55.600
31	B	165.800	6	155.1333	1.5346802	157.000	155.000	153.500	154.100	155.600	155.600
32	B	5.500	3	4.9900	0.0000000	4.990	4.990	4.990			
32	B	55.300	3	49.9000	0.0000019	49.900	49.900	49.900			
32	B	165.800	3	149.8000	0.0000153	149.800	149.800	149.800			
33	B	5.500	3	5.5400	0.0007000	5.510	5.550	5.560			
33	B	55.300	3	55.1033	0.0033398	55.070	55.170	55.070			
33	B	165.800	3	157.4000	0.1200104	157.200	157.200	157.800			

Table 10, continued. Silicate package A + AC.

SILICATE			A+AC					
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES		
35	B	5.500	3	5.9533	0.0161333	5.880	6.100	5.880
35	B	55.300	3	59.9000	0.2700024	59.600	59.600	60.500
35	B	165.800	3	161.5000	4.8399963	163.700	159.300	161.500
37	B	5.500	3	5.9100	0.2379000	6.460	5.740	5.530
37	B	55.300	3	57.4667	2.7033386	55.600	58.100	58.700
37	B	165.800	3	164.5667	76.0033417	154.500	169.600	169.600
40	B	5.500	3	5.5900	0.0027000	5.530	5.620	5.620
40	B	55.300	3	54.5400	0.0012035	54.580	54.520	54.520
40	B	165.800	3	162.6000	0.4800110	162.200	162.200	163.400
44	B	5.500	3	5.6000	0.0000000	5.600	5.600	5.600
44	B	55.300	3	54.6000	0.0000038	54.600	54.600	54.600
44	B	165.800	3	144.5000	0.0000000	144.500	144.500	144.500
7	Y	5.500	3	5.6000	0.0000000	5.600	5.600	5.600
7	Y	55.300	3	52.6000	5.0800056	50.000	53.800	54.000
7	Y	165.800	3	174.6667	2.0832977	173.000	175.500	175.500
19	Y	5.500	3	5.4667	0.0133334	5.400	5.400	5.600
19	Y	55.300	3	57.1333	0.1633339	57.500	56.700	57.200
19	Y	165.800	3	169.4000	0.0000000	169.400	169.400	169.400
26	Y	5.500	3	5.3333	0.3333333	6.000	5.000	5.000
26	Y	55.300	3	54.3333	0.3333340	54.000	55.000	54.000
26	Y	165.800	3	163.0000	0.0000000	163.000	163.000	163.000
28	Y	5.500	3	5.3333	0.0833333	5.500	5.500	5.000
28	Y	55.300	3	55.6667	0.3333302	56.000	55.000	56.000
28	Y	165.800	3	166.3333	0.3333588	166.000	166.000	167.000
5	A B	5.500	3	6.7000	0.0000000	6.700	6.700	6.700
5	A B	55.300	3	56.6000	0.0000057	56.600	56.600	56.600
5	A B	165.800	3	169.7000	0.0000000	169.700	169.700	169.700
18	A B	5.500	3	5.4733	0.0050334	5.550	5.460	5.410
18	A B	55.300	3	55.3333	0.0133362	55.200	55.400	55.400
18	A B	165.800	3	165.3333	0.3333588	166.000	165.000	165.000

Table 10, continued. Silicate package B + BC.

SILICATE			B+BC								
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES					
1	B	4.510	3	4.4000	0.0000000	4.400	4.400	4.400			
1	B	45.200	3	46.7333	0.0033379	46.700	46.700	46.800			
1	B	135.600	2	142.9000	0.0200043	142.800	143.000				
2	B	4.510	3	4.5300	0.0000000	4.530	4.530	4.530			
2	B	45.200	3	46.0000	0.0000000	46.000	46.000	46.000			
2	B	135.600	3	134.6000	0.0000305	134.600	134.600	134.600			
3	B	4.510	6	4.6983	0.0165767	4.660	4.620	4.710	4.520	4.680	4.800
3	B	45.200	6	46.5833	0.4776688	46.500	45.500	47.200	47.400	46.700	46.200
3	B	135.600	6	136.0167	5.7376953	135.300	138.400	139.500	133.900	135.300	133.700
6	B	4.510	3	4.2700	0.2623000	3.680	4.530	4.600			
11	B	4.510	3	4.7533	0.0002334	4.740	4.750	4.770			
11	B	45.200	3	45.7433	0.1497383	45.530	45.510	46.190			
11	B	135.600	3	134.6000	1.3900146	135.900	134.300	133.600			
16	B	4.510	6	4.1817	0.0104167	4.330	4.230	4.230	4.040	4.130	4.130
16	B	45.200	6	42.3400	0.3379211	42.020	41.540	42.020	42.980	42.500	42.980
16	B	135.600	6	117.9667	2.2426880	117.000	119.900	119.900	117.000	117.000	117.000
17	B	4.510	3	3.9400	0.0021000	3.950	3.980	3.890			
17	B	45.200	3	47.7333	0.0133381	47.800	47.800	47.600			
17	B	135.600	3	144.5333	0.0233459	144.700	144.500	144.400			
23	B	4.510	3	4.5000	0.0000000	4.500	4.500	4.500			
23	B	45.200	3	46.4000	0.0000038	46.400	46.400	46.400			
23	B	135.600	3	138.0667	0.2133636	137.800	137.800	138.600			
24	B	4.510	3	4.5000	0.0000000	4.500	4.500	4.500			
30	B	4.510	3	4.8667	0.0161333	4.940	4.720	4.940			
30	B	45.200	3	45.3200	0.0675011	45.170	45.620	45.170			
30	B	135.600	3	124.4667	0.2033539	124.900	124.500	124.000			
34	B	4.510	3	4.3767	0.0066334	4.320	4.470	4.340			
34	B	45.200	3	45.9333	0.0058346	45.850	45.950	46.000			
34	B	135.600	3	130.8667	0.2533722	130.800	131.400	130.400			
36	B	4.510	3	4.9267	0.0081333	4.920	4.840	5.020			
36	B	45.200	3	48.2667	0.0033360	48.200	48.300	48.300			
36	B	135.600	3	150.0000	0.0000000	150.000	150.000	150.000			
38	B	4.510	3	4.5600	0.0027000	4.530	4.620	4.530			
38	B	45.200	3	42.7667	0.0233355	42.600	42.900	42.800			
38	B	135.600	3	119.0000	1.0000000	118.000	119.000	120.000			
39	B	4.510	3	4.5333	0.0033334	4.600	4.500	4.500			
39	B	45.200	3	46.0667	0.0033340	46.100	46.000	46.100			
39	B	135.600	3	135.5333	0.0633392	135.300	135.500	135.800			

Table 10, continued. Silicate package B + BC.

SILICATE			B+BC								
LAB	MET	T. VAL	N	MEAN	VARIANCE	OBSERVED VALUES					
43	B	4.510	3	4.2333	0.0133333	4.300	4.300	4.100			
43	B	45.200	3	44.5000	0.0000000	44.500	44.500	44.500			
43	B	135.600	3	129.4667	0.1233368	129.100	129.500	129.800			
45	B	4.510	3	4.7767	0.0560333	4.640	4.640	5.050			
45	B	45.200	9	46.5222	0.6619453	45.800	45.800	46.600	48.300	47.200	46.600
45	B	135.600	9	128.4444	0.6952820	129.600	129.300	129.400	128.300	127.800	128.700
4	Y	4.510	3	4.2800	0.0013000	4.290	4.240	4.310	46.300	45.800	46.300
4	Y	45.200	3	44.9333	1.2233324	44.800	43.900	46.100	127.500	127.500	127.900
4	Y	135.600	3	135.3667	5.7633667	133.300	138.000	134.800			
6	Y	4.510	3	4.1667	0.1733334	3.700	4.500	4.300			
6	Y	45.200	3	46.6000	0.0400047	46.600	46.400	46.800			
6	Y	135.600	2	140.6000	0.0800018	140.400	140.800				
24	Y	45.200	3	42.0000	0.0000000	42.000	42.000	42.000			
24	Y	135.600	3	133.0000	0.0000000	133.000	133.000	133.000			
27	Y	4.510	3	4.8000	0.2100000	4.400	5.300	4.700			
27	Y	45.200	3	44.8000	0.0299778	44.900	44.600	44.900			
27	Y	135.600	3	134.6667	0.3333206	135.000	135.000	134.000			
20	A B	4.510	6	4.2700	0.0074000	4.120	4.250	4.250	4.350	4.350	4.300
20	A B	45.200	5	45.7600	0.0042515	45.800	45.750	45.800	45.800	45.650	
20	A B	135.600	5	126.7200	0.9719849	126.000	127.800	126.000	127.800	126.000	
41	A B	4.510	3	4.6300	0.0048000	4.550	4.670	4.670			
41	A B	45.200	3	48.7100	0.0000038	48.710	48.710	48.710			
41	A B	135.600	3	145.6667	0.0133820	145.600	145.800	145.600			

Indication of spine colours

Liaison Committee Reports	Red
Reports of Advisory Committee on Marine Pollution	Yellow
Fish Assessment Reports	Grey
Pollution Studies	Green
Others	Black

