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Ödsmål, Kville sn, Bohuslän

Hällristning
Fiskare från
bronsåldern

Rock carving
Bronze age
fishermen



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THE BALTIC ENTRANCE PROJECT:
Methods and Equipment. Quality of Measurements.
by
Staffan Löbf and Bodil Thorstensson

February 1980

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 TITELBLAD-RAPPORTER
3 Datum
1980-01-03
4 Ärendebeteckning(Diari nr)

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Denna sida får kopieras

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18 Sammanfattning av projektet/rapporten (ange gärna målsättning, metod, teknik resultat m m) <p>The hydrographic conditions in northern Kattegat were investigated in a project "The Baltic Entrance Project: the GF-section". In this paper information about all methods used for the analyses together with a short description of the equipment are presented. There is an attempt to verify the quality of measurements. In this discussion some statistical features are used and connected with this "the weak points" and source of errors within the analyses are mentioned.</p> <p>De hydrografiska förhållandena i norra Kattegatt har undersökts i ett projekt betitlat "The Baltic Entrance Project: the GF-Section". I detta papper ges information om alla metoder som använts för analyserna tillsammans med en kort beskrivning av utrustningen. Det görs ett försök att verifiera mätkvaliteten. I denna diskussion används några statistiska begrepp i relation till svaga punkter och felkällor inom analyserna.</p>					
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**THE BALTIC ENTRANCE PROJECT : Methods and
Equipment. Quality of Measurements.**

by

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Abstract.

The hydrographical conditions in northern Kattegat were investigated in a project "The Baltic Entrance Project: the GF-section". In this paper information about all methods used for the analyses together with a short description of the equipment are presented. There is an attempt to verify the quality of measurements. In this discussion some statistical features are used and connected with this "the weak points" and source of errors within the analyses are mentioned.

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1. Introduction

The project started in July 1974. The aim was to calculate the balance of water and material in northern Kattegat.

Ten stations were chosen between Göteborg and Frederikshavn (see table 1 and the map for positions). They were designated GF 1 - 10. The two stations GF-4 and GF-9 are the traditional stations SW Vinga and E Frederikshavn respectively.

The first expedition was carried out in August 1974. Until December 1977 75 cruises were made, i.e. roughly 20 a year. The vessel used was the "Ulla Rinman" (l.o.a. 24 m) belonging to the Swedish Sea Rescue Institution. Some expeditions were performed with research vessels belonging to the National Board of Fisheries - Sweden. The rescue vessel was equipped with some special arrangements for water sampling. The scientific staff consisted of three persons.

Mostly the stations were visited in the direction from Göteborg to Frederikshavn without abruption. When arriving at a station the first measure to be taken was laying out the Haamer current meters. Then water was sampled by means of reversing Knudsen bottles at the standard hydrographical depths. An isolated Knudsen sampler was used to collect the water just above the sediment. Thereafter the bathythermograph was lowered and a continuous temperature recording was obtained. If the station was included in the biology sampling program phyto- and zoo- plankton hauls were performed.

Before leaving the station the current meters were recovered and the capsules were read.

During the cruise from one station to the next the reversing thermometers were read and the water sampled was divided into different test tubes and bottles. In some cases reagents were added immediately. All analyses were performed at the laboratory ashore the subsequent day. The NO_2 - and NO_3 -samples were conserved with HgCl_2 , and TOC-samples with H_2SO_4 .

The quality of data is dependent on both sampling and on the analytical methods used. When choosing methods specificity, accuracy, precision and sensitivity have to be considered.

The methods used by the GF-project have generally been those described in the New Baltic Manual (hereafter referenced as NBM). They are almost identical with the methods used for standard hydrographical work at the laboratory (minor modifications have sometimes been introduced). However, the accuracy of each method, defined as the degree of agreement between the measured value and the true value, has so far not been established for this particular laboratory. Since it is very difficult to determine the true value of hydrographical parameters under field conditions, discussions on the reliability of chemical analyses are sometimes restricted to e.g. precision. Hence it was considered too great a task to establish the accuracy of the chemical analyses in the GF-project. Instead, our effort was concentrated on a study of the precision of the methods.

The standard deviation (SD) is frequently used as a measure of the precision of a method. Relative standard deviation, also called coefficient of variation (CV), is the standard deviation put in relation to the mean

value (MV) of a set of N independent measurements. The tables in this chapter will beside SD, CV, MV and N also display the maximum value (MAX), the minimum value (MIN) and their difference (DIFF) for each set of measurements.

Wet chemical methods have been used for the analyses of nutrients. When evaluating the precision of these methods certain limits for CV are sometimes used: up to 10 % is considered good, 10 - 20 % acceptable, 20 - 50 % doubtful and 50 % or more is unacceptable. The figures should, of course, be used only as a guide. Fonselius (1974) vindicates that Baltic surface phosphate values "should be reported with an accuracy of not more than around ± 20 % standard deviation". However, these data were computed from a time series at an anchor station. - When dealing with samples with concentrations close to the detection limit the standard deviation and the coefficient of variation increase rapidly. The above mentioned limits are then, of course, no longer applicable.

The results presented in this chapter derive from a group of data obtained at regular hydrographic expeditions. From each serial water sampler a large sample (nutrients only) was taken at the same occasion as the ordinary samples for the hydrographic series. Because of the large amount of water needed for 10 - 20 subsamples for salinity and oxygen determinations a special sampler (Hydro Bios 5 1) dedicated for this purpose only was employed.

2. Temperature T.

Methods and Equipment.

A thermometer is fixed to each sampler. The kind of thermometer used is a protected reversing thermometer. The temperature was determined to one hundred of a centigrade.

Quality.

The thermometers used were delivered (various years before the GF-project started) by the manufacturer with a certificate of precision. Right before the measurements started the thermometers were recalibrated. There were rather great changes, max. 0.1 °C, from the last calibration for reasons which are not clear. Most important is the aging factor. Let us assume this error to be 0.04 °C at its most. The reading adds approximately 0.01 °C, and so does the correction procedure. We then arrive at a maximum error of ± 0.06 degrees Celsius.

3. Salinity S.

Methods and Equipment.

The conductivity of the samples was measured by means of a laboratory salinometer manufactured by Plessey Environmental System, U.S., model 6230 N.

Quality.

For the salinity samples a bottle containing approximately 170 ml was used. To decrease the carry over effect between samples of markedly different salinities. The routine samples are analysed in the order surface to bottom, bottom to surface and so on. This is very important, especially when using bottles of this size since the amount of water is generally not sufficient to rinse the measuring cell completely twice. Due to the carry over effect sometimes two sets of data exist in the tables describing the results of precision investigations with N and N-1 samples respectively.

In these cases it was observed that the first sample in the series was contaminated by the preceding one. Therefore, a second statistical calculation has been performed where the first value has been excluded (table 2).

Most manufacturers of the type of salinometer used in this project claim a reproducibility of $\pm 0.002 - 0.003$ ‰ S. As revealed by table 2 this instrument precision can be obtained when the measurements are carefully performed with sufficient rinsing between samples of different salinities. However, intercalibrations have shown that the comparability of salinity measurements performed with different instruments, even of the same type, is much worse than the reproducibility of one and the same instrument. A standard deviation of ± 0.01 ‰ S is therefore claimed as a more realistic value (Grasshoff 1976).

Due to the influence of the carbonate system, the accuracy of salinity measurements cannot be better than ± 0.02 ‰ S. This should be kept in mind when evaluating data from conductivity measurements. Within the period of the project the old laboratory salinometer had to be replaced by a new one. So two different instruments, but of the same type, have been used for the salinity determinations in this project. However, all values in table 2 were recorded with one and the same instrument. Therefore, ± 0.01 ‰ S is stated as the precision of the GF-project salinity determinations.

4. Oxygen O₂

Methods and Equipment.

This analysis was made according to the common Winkler method, (modified by Carritt and Carpenter, see NBM).

Quality.

In order to examine the quality of the oxygen determina-

tions 10 (in some cases 20) subsamples were drawn from a 5 l water-sampler. Titration was then carried out according to the standard procedure. Several tests with water containing different amounts of oxygen were performed. One would perhaps suspect that due to oxygenation during the subsampling procedure the samples at the end of a series could yield somewhat higher oxygen values. However, this was not observed.

Intercalibration exercises with 5 participating laboratories gave a precision expressed as standard deviation of ± 0.03 ml/l (Fonselius 1966, 1968). In literature, the reproducibility is often stated to be as good as ± 0.02 ml/l for oxygen contents of less than 2 ml/l and ± 0.04 ml/l for oxygen contents above 2 ml/l.

Table 3 suggest that a skilled analyst sometimes can obtain a standard deviation of ± 0.02 ml/l for samples richer in oxygen than 2 ml/l (column 4 and 7). As in all titration analyses, the personal experience and fitness is of great importance to the precision. Inability to detect the correct endpoint of the titration will e.g. give values with great scattering.

The major contribution to the systematic error is to be found in the sampling procedure. This seems to be worth special attention especially when working with samples of low oxygen contents (Grasshoff 1976).

5. Phosphorus.

a) Phosphate phosphorus PO₄-P Methods and Equipment.

The samples were determined by a method which is a modification of the method by Murphy and Riley. The reagents are the same as for total phosphorus (see paragraph b). 0.7 ml of each reagent is added, molybdate reagent before the ascorbic acid.

Quality.

Table 4 shows 17 sets of phosphate measurements in the range 0 - 1 µgat/l. Even if SD is low throughout the series (generally ± 0.04 µgat/l), concentrations below 0.10-0.12 µgat/l will occasionally give rather dispersed values. It has been proved that an insufficient cleaning of the test tubes might cause a considerable scattering in a set of replicate samples (Dahlin, pers. com.). The general trend is that most samples in a set is fairly well gathered in a cluster around a central value but sometimes the material is biased by one or two strongly deviating measurements. But SD remains mostly at a low level because these values are relatively scarce.

Hydrographers consider the phosphate analysis to be trouble-free, simple and accurate. Koroleff and Palmork (1972) give a relative error of accuracy of ± 15 % at the 0.2 µgat/l-level and ± 5 % at the 0.9 µgat/l-level. This corresponds to ± 0.03 and ± 0.05 µgat/l respectively. The limit of detection is 0.02 µgat/l.

b) Total phosphorus TOT-P Methods and Equipment.

A modification of the method described in NBM has been used. The following parts differ: sample volume is 25 ml; the ascorbic acid reagent contains only 70 g ascorbic acid per litre together with 0.3 g EDTA and 6 ml formic

acid; 0.7 ml of each reagent is added, ascorbic acid before the molybdate reagent. The oxidation has been made in closed bottles at overpressure in a kitchen pressure cooker.

Quality.

From the sets of measurements displayed in table 5 the conclusion might be drawn that the precision, expressed as standard deviation, is almost as good as for PO_4 -P. However, by experience analysts know that this method may occasionally give rather peculiar results. Hence special attention has been paid to the checking of total phosphorus data. Despite this some of the data might still be questionable.

The range of a set of data is usually not recommended as a measure of precision. However, if a set consists of only a few measurements the range might be of some use. In table 5 some extremely great differences is encountered (e.g. columns 2 and 7). NBM says that "the precision is very good and the reproducibility is in most cases better than 0.7 μ g/l (0.02 μ gat/l)". However, the analysing procedure contains a rather subjective step: adding of the oxidizing agent by means of a spatula. The spatula, made of perspex contains a cavity. This cavity is filled with $K_2S_2O_8$ and the amount is adjusted by drawing a steel spatula along the perspex surface. In this way the excess of the oxidizing agent is removed. It is important that the amount of peroxydisulphate is kept constant from time to time since this influences pH and in this way the colour development.

Summing up the precision of the total phosphorus measurements is not as good as it is for phosphate phosphorus. Taking into consideration the variations found at intercalibration exercises and tests at the laboratory an average precision expressed as standard deviation of approximately ± 0.06 μ gat/l is probably a realistic choice.

6. Nitrogen.

a) Ammonia NH₄-N.

Methods and Equipment.

The method used has been the one described by Folke Koroleff (see NBM), where blue indophenol is formed in the presence of ammonia. The reagent called B has been exchanged to a modified variant, described under "Remarks" in NBM. Further on in this manual there is a description of the buffer used for the samples to avoid precipitate.

Quality.

When performing ammonia analyses cleanliness and protection of the samples from sources liable to cause contamination is of utmost importance.

As was the case with total phosphorus measurements ammonia analyses will sometimes give some deviating values in a set of replicate samples. Table 6 gives an example of this. Generally SD is in the order of $\pm 0.02 - 0.03 \mu\text{gat/l}$. In a couple of sets some extreme values increase the range (DIFF) and therefore SD and CV as well.

NBM states a reproducibility better than $\pm 5 \%$ in routine work. Dahl (1973) presents results from an intercalibration exercise which gave $\text{CV} = \pm 4.8 \%$. These figures seem to be a bit low according to experience at the Institute of Hydrographic Research. A representative value of CV is rather found in the range $\pm 5 - 10 \%$. The limit of detection is $0.02 \mu\text{gat/l}$.

b) Nitrite NO₂-N.

Methods and Equipment.

This analysis was performed according to NBM with sulphanilamide and diamine dihydrochloride giving a red azo-product that was measured photometrically.

Quality.

The method for the determination of nitrite seems to be the most accurate one among those dealing with nutrients containing nitrogen. As can be seen from the few examples in table 7 the reproducibility is extremely good. Seldom does the difference between the highest and the lowest value in a set of ten exceed $\pm 0.02 \mu\text{g}/\text{l}$ which in fact is the inaccuracy in the instrument display. Grasshoff (1976) claims a reproducibility of $\pm 0.02 \mu\text{g}/\text{l}$. The limit of detection is $0.02 \mu\text{g}/\text{l}$.

However, field comparisons suggest that the good reproducibility often is of little help when working practically. Due to the inaccuracy in the depth determination when using conventional sampling methods it is very difficult to collect water from the narrow layers where nitrite compounds often accumulate. Figure 1 and 2 show the results of a field comparison between two research vessels with separate samplings (Fonselius, 1979).

- Another problem is the very low concentrations close to the detection limit that are relatively frequent.

c) Nitrate $\text{NO}_3\text{-N}$
Methods and Equipment.

The treatment of these samples followed NBM. In those cases where there are alternative ways of performance the following alternative was chosen: the reducers used were filled with amalgamated cadmium granules; ammonium chloride-ammonia solution was used to buffer the samples, called buffer B in the manual.

Quality.

A critical factor in nitrate determinations is the condition of the reducers. A reductor in good shape should give a deviation between duplicate samples of not more than $\pm 0.1 \mu\text{g}/\text{l}$ in the range 0 - 5 $\mu\text{g}/\text{l}$ and $\pm 0.2 \mu\text{g}/\text{l}$ in the range 5 - 10 $\mu\text{g}/\text{l}$ (Grasshoff 1976). In non-automated systems the U-shaped type has

proved to be the most suitable one when using amalgamated cadmium. The reason for this is the need for a reproducible flow speed.

The ten samples in each set displayed in table 8.1 have all been run on different reducers. CV is generally $\pm 3\%$ or better. Grasshoff (1976) claims that "if no systematic sampling errors are involved, the relative accuracy of the nitrate determinations can be assumed to be $\pm 3\%$ in the range between 0 to 3 $\mu\text{gat NO}_3\text{-N}\cdot\text{l}^{-1}$ ".

Nitrate data from the GF-project have been reported to the first decimal place. If the reducers are freshly calibrated and found to give a high yield the second decimal place could be relevant. Due to the project design calibrations could not be performed before every single expedition. Therefore the relevancy of the second decimal was considered questionable. The lowest detectable amount of nitrate is 0.1 $\mu\text{gat/l}$.

d) Total nitrogen TDI-N
Methods and Equipment.

In NBM there are some alternative treatments; the samples of 10 ml were boiled in closed bottles at overpressure together with 15 ml of the oxidation mixture. The bottles used were 50 ml "Sovirel" bottles closed by stoppers of polypropylen. After boiling 1.5 ml 0.3 N hydrochloric acid was added (see under "Comments" in NBM). After cooling, the samples were buffered with 2 ml borate buffer. The following treatment on reducers and addition of reagents was the same as for nitrate.

Quality.

No tests have been performed in order to establish the precision of this total nitrogen analysis. The method employed is known to be of a rather low precision.

The oxidation of phosphorus compounds in sea-water to phosphate is supposed to be almost quantitative when using peroxydisulphate as the oxidizing agent and boiling at overpressure. This seems not to be the case with nitrogen compounds. Furthermore, when adding the alkaline solution containing the oxidant, ammonia is formed and liberated. An additional loss might be caused by the formation of gaseous nitrogen during the oxidation process. Hence, total nitrogen data from the GF-project are reported as whole numbers only. The new method by Koroleff, where both total nitrogen and total phosphorus are analysed, has been tested by Valderrama (to be published).

7. Total Organic Carbon TOC

Methods and Equipment.

The analyses were made according to Menzel and Vaccaro at the Institution of Marine Botany at University of Lund.

Quality.

Data of total amount of organic carbon from the GF-project have been reported to the first decimal place. Within a series of 5 samples the differences range to 0.10 - 0.12 mg/l. This will give a reliability of the chemical analysis of ± 0.06 mg/l. Most samples lie in the range of 2 - 3 mg C/l. Extreme values like 0.6 and 9.3 mg C/l are seldomly found.

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natural waters (to be published).

Table 1.

Positions of
the section Göteborg - Frederikshavn.

GF	1	N 57°34.7'	E 11°51.5'
	2	34.45'	48.5'
	3	33.7'	40.0'
	4	33.0'	31.5'
	5	32.3'	26.0'
	6	32.0'	19.5'
	7	30.3'	8.5'
	8	27.9'	10°54.0'
	9	26.0'	42.5'
	10	25.0'	35.7'

SALINITY 4 S

	1 a	1 b	2 a	2 b	3	4 a	4 b	5	6	7
MAX	19.759	19.745	19.877	19.862	20.445	30.152	30.152	31.779	32.559	33.954
MIN	19.740	19.740	19.859	19.859	20.443	30.122	30.135	31.773	32.553	33.948
DIFF	0.019	0.005	0.018	0.003	0.002	0.030	0.017	0.006	0.006	0.006
MV	19.744	19.742	19.863	19.860	20.444	30.140	30.142	31.776	32.555	33.951
SD	0.006	0.002	0.008	0.001	0.001	0.008	0.005	0.002	0.002	0.002
CV %	0.030	0.010	0.040	0.005	0.005	0.027	0.017	0.006	0.006	0.006
N	10	9	5	4	8	10	9	10	10	10
	-- same set --	-- same set --	-- same set --	-- same set --		-- same set --				

" -- same set -- " indicates a set of samples where it has been obvious that the first sample was contaminated by the preceding one by the carry over effect in the measuring cell. This value has been excluded and a second calculation performed.

Table 3.

OXYGEN - O₂ ml/l

	I	II	III	
	N=10	9	20	
MAX	0.28	0.32	4.56	
MIN	0.18	0.18	4.48	
DIFF	0.10	0.14	0.08	
M	0.23	0.22	4.51	
SD	0.03	0.04	0.03	
CV %	14	18	0.66	
	IV	V	VI	
	N=10	9	10	
MAX	4.76	5.02	5.04	
MIN	4.69	4.93	4.98	
DIFF	0.07	0.09	0.06	
M	4.71	4.97	5.02	
SD	0.02	0.03	0.02	
CV %	0.42	0.65	0.35	
	VII	VIII	IX	X
	N=10	10	20	10
MAX	5.07	5.93	5.96	6.68
MIN	5.01	5.81	5.81	6.57
DIFF	0.06	0.12	0.15	0.11
M	5.05	5.87	5.89	6.61
SD	0.02	0.04	0.03	0.03
CV %	0.40	0.68	0.57	0.45

Table 4.

PHOSPHATE PHOSPHORUS $\mu\text{g}/\text{l}$ N=10							
	1	2	3	4	5	6	
MAX	0.04	0.05	0.07	0.07	0.12	0.13	
MIN	0.01	0.02	0.02	0.04	0.05	0.11	
DIFF	0.03	0.03	0.05	0.03	0.07	0.02	
MV	0.03	0.04	0.05	0.03	0.07	0.12	
SD	0.01	0.01	0.02	0.01	0.02	0.01	
CV %	40	34	31	18	29	8	
	7	8	9	10	11	12	
MAX	0.14	0.25	0.29	0.31	0.38	0.46	
MIN	0.12	0.18	0.22	0.22	0.32	0.37	
DIFF	0.02	0.07	0.07	0.09	0.06	0.09	
MV	0.13	0.21	0.25	0.27	0.34	0.42	
SD	0.01	0.02	0.02	0.04	0.02	0.02	
CV %	6	11	9	15	5	7	
	13	14	15	16	17		
MAX	0.53	0.58	0.65	0.98	1.02		
MIN	0.49	0.51	0.56	0.81	0.98		
DIFF	0.04	0.07	0.09	0.17	0.04		
MV	0.51	0.55	0.58	0.90	1.00		
SD	0.01	0.02	0.03	0.06	0.01		
CV %	3	4	5	7	2		

Table 5.

TOTAL PHOSPHORUS $\mu\text{gat}/1$ N=10

	1	2	3	4	5
MAX	0.38	0.57	0.64	0.79	0.83
MIN	0.28	0.31	0.58	0.67	0.74
DIFF	0.10	0.26	0.06	0.12	0.09
MV	0.34	0.42	0.61	0.72	0.80
SD	0.03	0.07	0.02	0.04	0.03
CV %	9	17	3	5	4
	6	7	8	9	
MAX	0.89	0.90	1.16	1.24	
MIN	0.75	0.63	1.10	1.10	
DIFF	0.14	0.27	0.06	0.14	
MV	0.77	0.72	1.14	1.15	
SD	0.04	0.07	0.02	0.04	
CV %	5	10	2	4	

Table 6.

AMMONIA NITROGEN $\mu\text{g}/\text{l}$					
	1	2	3	4	5
MAX	0.40	0.42	0.43	0.44	0.62
MIN	0.26	0.35	0.34	0.34	0.26
DIFF	0.14	0.07	0.09	0.10	0.36
MV	0.28	0.39	0.38	0.33	0.33
SD	0.05	0.02	0.03	0.03	0.07
CV %	16	5	7	9	23
N	10	10	20	20	20

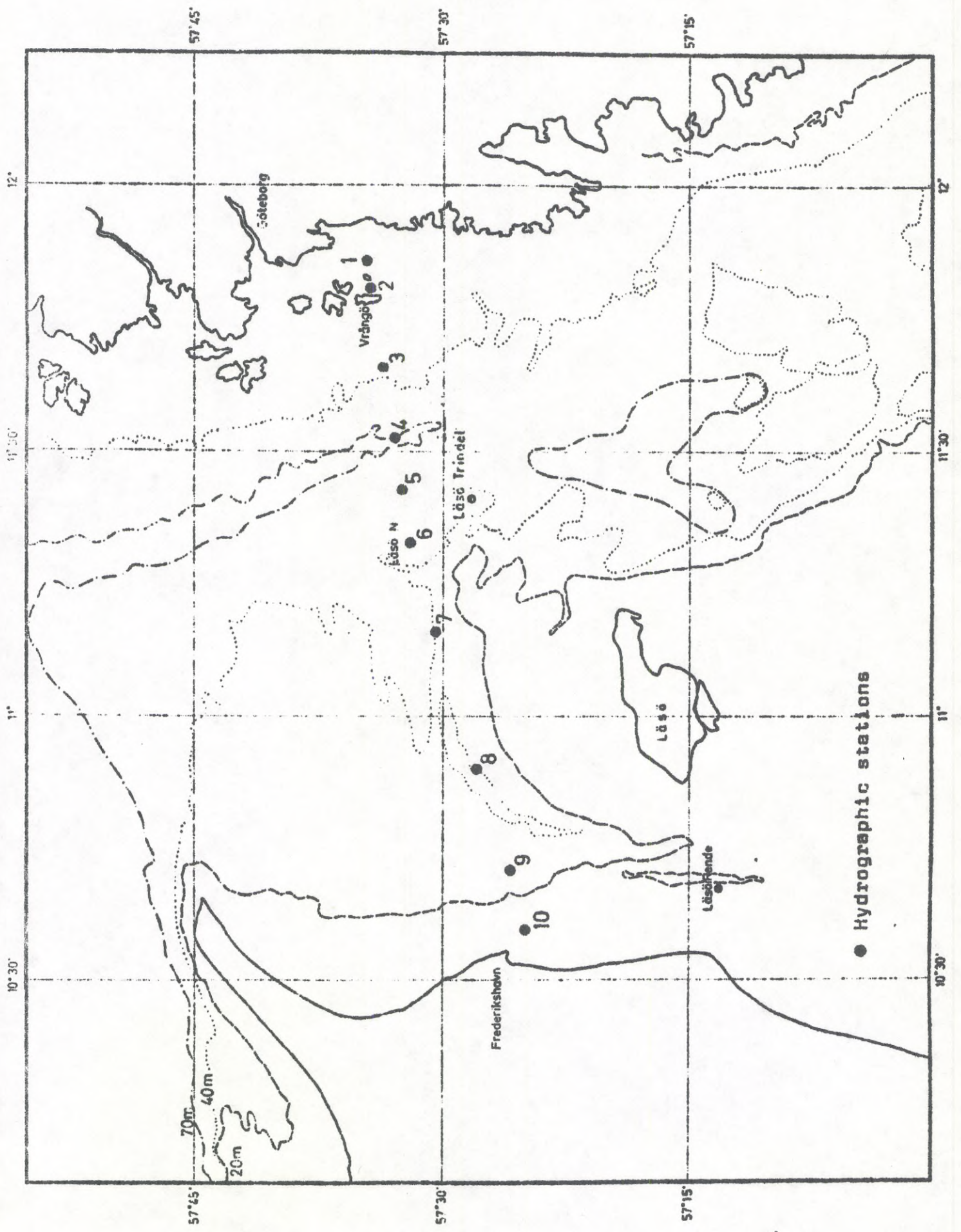
Table 7.

NITRITE NITROGEN $\mu\text{g}/\text{l}$ N=10					
	1	2	3	4	5
MAX	0.13	0.14	0.16	0.36	0.57
MIN	0.09	0.13	0.16	0.35	0.55
DIFF	0.04	0.01	0	0.01	0.02
MV	0.11	0.13	0.16	0.36	0.56
SD	0.02	0.003		0.004	0.006
CV %	14	3		1	1

Table 8.

NITRATE NITROGEN $\mu\text{gat}/1$ N=10

	1	2	3	4	5
MAX	1.9	1.9	2.2	2.2	3.5
MIN	1.8	1.8	2.0	2.0	3.3
DIFF	0.1	0.1	0.2	0.2	0.2
MV	1.8	1.9	2.1	2.1	3.4
SD	0.05	0.05	0.06	0.07	0.06
CV %	3	3	3	3	2
	6	7	8	9	
MAX	3.7	3.8	6.6	11.4	
MIN	3.3	3.6	6.0	11.1	
DIFF	0.4	0.2	0.6	0.3	
MV	3.4	3.7	6.2	11.3	
SD	0.10	0.06	0.18	0.15	
CV %	4	2	3	1	



Penck - Argos NO₂ µgat/l

Linear regression

Number of points 35

x-values assumed to be independent and y-values dependent

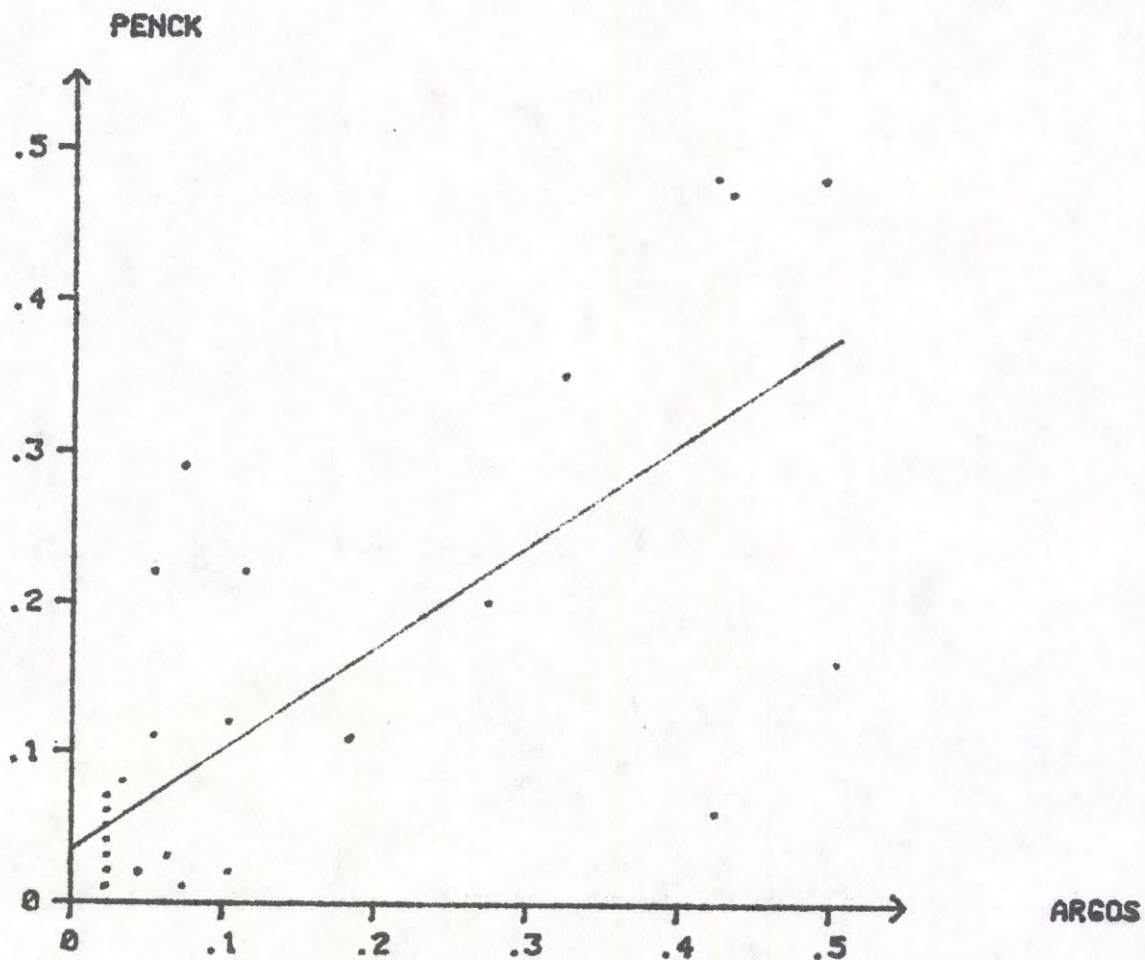
The equation of the line: $Y=A+B \times X$.

Interception of the line with y-axis = $A = 0.04 \pm 0.10$
(with 95% probability)

Inclination of the line = $B = 0.67 \pm 0.20$
(with 95% probability)

Corr. coeff. 0.751304

INTERCOMPARISON NITRITE (UGAT/L) BOSEX



Poseidon - Argos NO_2 $\mu\text{g}/\text{l}$ Linear regression

Number of points 88

x-values assumed to be independent and y-values dependent

The equation of the line: $Y=A+B \times X$ Interception of the line with y-axis = $A = 0.03 \pm 0.07$
(with 95% probability)Inclination of the line = $B = 0.69 \pm 0.17$
(with 95% probability)

Corr. coeff. 0.648386

INTERCOMPARISON NITRITE ($\mu\text{GAT}/\text{L}$) BOSEX

POSEIDON

