

COMMISSION OF THE EUROPEAN COMMUNITIES

Environment and quality of life

**A EUROPEAN COMMUNITY STUDY
ON THE DETERMINATION OF
CYANIDES, PHENOLS AND HYDROCARBONS
IN SURFACE WATER**

Report of a Working Group of Experts



Directorate-General
for Social Affairs

Health Protection
Directorate

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A EUROPEAN COMMUNITY STUDY ON THE DETERMINATION OF CYANIDES, PHENOLS AND HYDROCARBONS IN SURFACE WATER

Report of a Working Group of Experts

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(Commission of the European Communities)

Directorate-General for Social Affairs

Health Protection Directorate

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1. I n t r o d u c t i o n

The Commission of the European Communities, Health Protection Directorate, Luxembourg, in January 1974, suggested a collaborative study in the working group 'Relatively unstable organic compounds' to detect free cyanides, phenols and hydrocarbons in surface water. For this no single method was to be used, but different methods chosen by the participants were to be tested for comparability of the results. The aim of this work was a preliminary experiment for quality control of analytic measurement rather than a collaborative study with a standard procedure, so that the information from the European Community relating to environmental pollution by these above mentioned compounds (see H. Mühleck: Occurrence of Phenols and Hydrocarbons in the Environment; report EC, Luxembourg, in press) could be evaluated. The Health Protection Directorate collaborated with the Environment and Consumer Protection Service in this study and in April 1974 the preliminary plans were discussed between them and the Institut für Wasser-, Boden- und Lufthygiene des Bundesgesundheitsamtes, Berlin. An explanatory note (see appendix 10.1) was sent to interested laboratories. Thirtyfour laboratories from different European countries expressed their interest (Fig. 9.1).

The schedule was revised:

1. Delivery of the samples by 13th August, 1974
2. Results sent back by 5th October, 1974
3. A meeting of the participating laboratories in Luxembourg on the 4th - 5th November, 1974, for a final discussion

2. T e c h n i c a l d e t a i l s

Technical details were as follows:

The water samples were to be examined for their content of free cyanides, phenols and hydrocarbons by any chosen method, and to ensure that enough sample material was available, for those methods not known to us to begin with, 3 samples (each 2 litres) of A, of B and of C were provided. For shipping purposes 2-litre red wine bottles seemed appropriate. Preliminary studies showed that the samples were not affected by absorption by the corks or by the glass. The absorption was less than 1 % (see 3.1.).

As basic material a heavily polluted 'surface water' was chosen, so that the analytical quality control was carried out under conditions approximately corresponding to those met while monitoring surface or waste water.

The samples were prepared in the following way: 700 litres of surface water were taken from a 'river' and kept in a fiberglass container for 12 hours to allow the main part of the suspended matter to settle. 550 litres of the supernatant sludge water were pumped into a second tank, diluted with 250 litres of tap water and adjusted to pH 11 with NaOH to stabilize the sample. Most of the calcium and magnesium salts precipitated from the surface water were removed by filtering twice through gravel filters. The remainder precipitated during transportation.

Samples A and B were bottled from this solution and not taken from two different surface waters, as originally intended. The identity of A and B was an additional advantage for statistical evaluation, because the individual values of samples A and B

could be compared with each other.

Sample C was prepared from 250 litres of the original sample material adding the following compounds:

- (1) Potassium cyanide to a final concentration of 3.9 mg CN⁻ per litre sample
- (2) 2,6 - dichlorophenol to a final concentration of 1 mg phenol per litre sample
- (3) octahydrophenanthrene to a final concentration of 1 mg C per litre sample

After these additions and five hours of intensive mixing sample C was bottled.

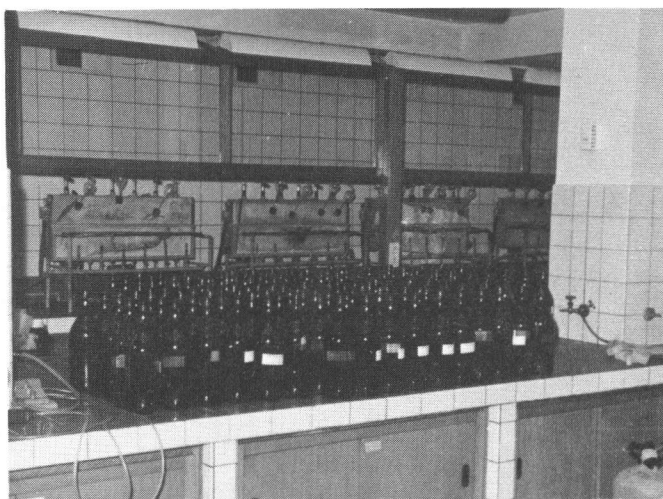
The finished samples were air-freighted to the participating laboratories on the 30th of August, 1974. Minimum-maximum recording thermometers were packed in the shipping boxes because temperature variations were expected; thus a higher temperature during shipment could be determined. This showed the highest temperatures occurred not only on the transportation south to Italy but also to the middle of Europe (all Belgian and one French laboratory). In most other cases the measured maximum temperature remained below 30° C. (Table 8.1.).

The duration of transport varied, in some cases the journey took as long as 18 days (Table 8.1.). This (and other reasons) caused a delay in the scheduled October 5th, 1974, return of the results, so that only one third of the results were received on that date by the rapporteur. This made it difficult to give the final evaluations to the participants at the scheduled meeting on the 4th and 5th November, 1974, in Luxembourg (see appendix 10.6.). The results and details of the analytical

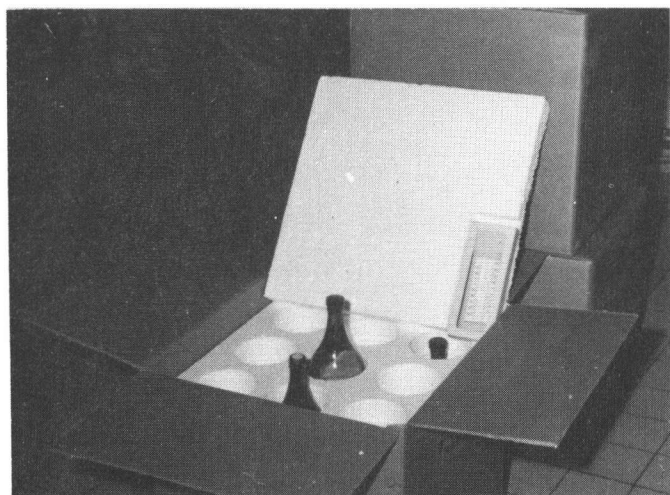
Technical Preparations



I



II



III



IV

- I. Bottling of samples from the container
- II. Samples before packing
- III. Sample boxes, filled with polystyrene for thermoisolation. At the right side of the lid the minimal-maximal thermometer for temperature measurement
- IV. Finished packages

methods that were not sent up to this time were compiled and the results found were discussed at this meeting.

3. S e l e c t e d p r e l i m i n a r y i n v e s t i g a t i o n s (carried out by the Ruhrverband und Ruhrtalsperrenverein, Essen)

3.1. Absorption behavior

Corks, bottles and precipitates of the samples were investigated for possible absorption of aliphatic hydrocarbons. The corks used to seal the bottles were shaken intensively for 20 hours in water (sample C) and the contents of the corks extracted with CCl_4 . Infra-red measurements showed that the total absorption of a cork was as much as 1 % of the total hydrocarbons present in a bottle. Taking into consideration the geometric ratio of the corks surface in contact with the sample material the results would not therefore be affected. In a further experiment using sample water C the hydrocarbons in the precipitates (mainly calcium and magnesium salts) were extracted with CCl_4 and the empty bottle was washed three times with CCl_4 . The combined extractions were analyzed for aliphatic hydrocarbons by the IR-method. No significant absorption by the precipitate or the glass was detected.

3.2. Time dependence

To control the possible decay of phenols in the course of time an aliquot of sample A was analyzed over a period of 20 days for the phenol content. The results are shown in Figure 9.6. A slight increase of the concentration could be detected by the 6th day after preparation, then a decrease of phenol concentration to the original value by the 20th day. The time dependence of the concentration of cyanides will be discussed in chapter 6.

4. General results

The results of the measurements of the individual participating laboratories are presented in tables 8.2. - 8.3. and figures 9.2. - 9.3. for cyanides and phenols. Some of the 10 participants determined the hydrocarbon content as total organic carbon (TOC), but the others determined only aliphatic hydrocarbons. These results are shown in tables 8.4. and figures 9.4. - 9.5. In the following the determination of TOC will be treated as an additional parameter along with aliphatic hydrocarbons.

As already mentioned results were received by the rapporteur after some delays. Even then, the required detailed description of the analytical methods used for the determination were, in many cases, inadequate making an exact interpretation of the data difficult. Therefore at the meeting in Luxembourg on the 4th and 5th of November, 1974, questionnaires were distributed to collect the informations necessary for evaluation (see appendix 10.4.). These informations on the analytical methods used by the participating laboratories in this comparative study are summarized in tables 8.5. - 8.8.

5. Statistical methods

Several statistical tests were made on the results from the individual laboratories. First, all the measurements of one sample were combined and tested for outliers, using the NALIMOV procedure *. Mean, standard deviation for single measurements, standard deviation for the mean and the 95 %-confidence

* Kaiser, R., and G. Gottschalk: Elementare Tests zur Beurteilung von Meßdaten, Hochschultaschenbücher Bd. 774, B.I.-Wissenschaftsverlag Mannheim 1972

interval were calculated excluding outliers.

In tables 8.9. these data are given. Outliers are identified by *. Additionally for each laboratory (or in case several methods were used, for each series of measurement), mean and standard deviation are given. Due to the large variation in the results of measurements additional tests for the variation of mean \bar{x} and standard deviations were made on the normally used range ($\bar{x} \pm 4s$) for identification of outliers. Only small changes resulted which were taken into consideration in the discussion of the measurement parameters in the following sections. The different values of mean and standard deviation are summarized in table 8.9. The individual measurements are not 'normally distributed' as shown by χ^2 -tests, even when outliers have been eliminated. The homogeneity of the data could not be proven by variance analysis. Regrouping of the individual measurements according to the analytical methods did not lead to better homogeneity. Test comparisons between samples A and B were made for all measurements as well as for each series of measurements from the individual laboratories.

6. D i s c u s s i o n

6.1. D e t e r m i n a t i o n o f c y a n i d e s

Of the 28 laboratories which determined cyanides only three did not distil the sample. 24 laboratories applied colorimetric measurement using the colour-reaction pyridine-pyrazolone (17 laboratories), pyridine-p-phenylenediamine (4 laboratories) or pyridine-barbituric acid (2 laboratories). Ion-selective electrodes were used by 4 laboratories and 3 carried out titrimetric determinations (see table 8.5.).

For all the different methods the results were spread over a wide range (figure 9.2.). For sample C, where the concentration was higher, the values are grouped more closely except for a few outliers (figure 9.7.). The trial to combine groups according to analytical methods did not lead to better results, i.e. for all methods the range seems to be high, even though the results from one individual laboratory agreed very well. A collaborative study organized in Germany last year showed differences of the same magnitude*.

Samples A and B, originally bottled from the same container, showed no significant difference in the mean which was 0,1 mg/l (standard deviation 0,07 mg/l) (see table 8.9.). When comparing the 29 series of measurements at the 1 % significance level 10 means were different, 15 not, 4 could not be compared statistically because each series was identical. The means of sample B were slightly higher than those of A in most cases (see Youden plot at figure 9.3.).

The mean of all determinations of sample C made in the first 25 days after sampling agrees with the theoretical concentration of 4 mg CN^- /l (original concentration plus spiking). This value lies within the 95 % confidence interval of the mean: $3,81 \pm 0,25$ mg/l (group CII in last table 8.9.). However when the determinations were made at a later date the measured values were lower (figure 9.6. - 9.7.).

An evaluation of the time dependence shows therefore a significant division into 2 groups:

* unpublished, quoted in "Der Spiegel", Nr. 44 (1974)

1. measurement after the 25th day
2. measurement before the 25th day after sampling.

Both groups in the last table 8.9. are identified by AI and AII etc. The concentration of cyanides evidently decreased with increasing age of the samples. Therefore it seems appropriate for future collaborative tests to set a time limit of 20 days for sampling, mailing and measurement. Further explanations for the variation of the results could not be found. One laboratory reported a calculating mistake for which the true value is lower by one order of magnitude (Lab. C).

6.2. Determination of phenols

By all but two laboratories, which use gas chromatography or thin-layer chromatography the phenol content was determined by colorimetric methods. The colour-reagent 4-amino-antipyrine was used by 26 laboratories, other colour-reagents by 4 laboratories. The samples were prepared for analysis mostly by distillation, but also by filtration or extraction (table 8.6.). The final evaluation did not show any significant differences caused by the different methods.

2,6-dichlorophenol had been used for spiking sample C when preparing the samples. This compound could have been detected only by gas chromatography or thin-layer chromatography. These methods, however, were only applied qualitatively by two laboratories. Using the colorimetric method, 2,6-dichlorophenol can be determined by the reaction with p-nitraniline up to 5 % only or, with 4-amino-antipyrine, up to 9 %. Therefore no difference could be expected in the measurements of sample A and B on one hand and sample C on the other.

The results of the determination of phenols were clearly grouped more closely together than those of cyanides (figure 9.3. and 9.7.). However, it must be recalled that the measured concentrations lay in a range which corresponded to that of a heavily polluted surface water.

No explanation could be found for the outlying results of some laboratories. The investigation on time dependence which had been carried out as one of the preliminary examinations (see section 3.2.) had shown slight changes of the phenol content during the first 10 days (figure 9.6.1.). When evaluating the results of all measurements by the participating laboratories no time dependence of the measured concentrations could be detected (see figure 9.6.2.). The mean values of samples A, B and C show - as was to be expected - no significant difference. The mean concentrations of phenols are 7,8 mg/l (standard deviation 1,0 mg/l) (see tables 8.9.). When comparing the means of 33 individual series of measurements of samples A and B only 4 were different at the 1 % significance level (see Youden plot at figure 9.3.).

6.3. Determination of total organic carbon

Instead of, or in addition to, the determination of aliphatic hydrocarbons 6 of the participating laboratories analyzed the samples for total organic carbon. For this they measured either the carbon dioxide, formed from the organic carbon, by an IR-detector (3 laboratories) or methane by a flame-ionisation-detector (3 laboratories) (see table 8.7.). The specific methods of measurement could not account for the differences of the individual results (see table 8.3.). Only

the values of one laboratory showed significant differences according to whether the samples had been mixed or centrifuged and decanted before measurement. The difference between the mean values of samples A and B is significant on the 1 % level. At the same significance level 3, out of 7 series of measurements, were different and 4 not. Due to the limited number of results any time dependence could not be evaluated.

6.4. Determination of aliphatic hydrocarbons

Determinations of the content of aliphatic hydrocarbons were carried out by 16 laboratories using the infra-red method. One laboratory determined the residue by gravimetry after acidification and extraction with petroleum ether; their measurements having the largest variation of single values. When determining the hydrocarbons by the IR-method the standard deviations of the single values were in some cases quite small (see figure 9.5.). A comparison of all measurement results showed large differences which could not be accounted for by different precleaning with and without absorption on florisil or Al_2O_3 . The significant difference of the means of samples A and B could not be explained. Even though sample B was bottled before sample A from the water container the concentrations found were lower in B than in A, the mean of B being 0,9 mgC/l compared to 1,9 mg C/l in A. The assumption that part of the hydrocarbons evaporated during the bottling procedure could explain why sample C showed the lowest concentrations, having been bottled 24 hours later. But in this case, the mean of sample A should also have been lower than that of B. Storage time effects, until the samples were

analysed, could not be detected because of the large variations in the results.

7. S u m m a r y

With the participation of 34 laboratories from 8 Member States of the European Community a preliminary collaborative study on the detection of cyanides, phenols and hydrocarbons in surface water was conducted. The samples were received by nearly all participants within 6 days of shipment, only the transport to 7 participants taking a longer time. With a few exceptions the variation in temperature during transport was relatively small.

Each participating laboratory chose the method of analyzing the samples. An appraisal of the particular methods could not be done.

In detail the evaluation showed that the determination of hydrocarbons and cyanides in surface water may lead to considerably different results. In higher concentration ranges ($> 1 \text{ mg CN}^-/\text{l}$) the results of the measurements of cyanide content were in better agreement. In the concentration range given in this study the determinations of phenol showed the smallest variation of all parameters studied. In general the results of the multiple determinations of all parameters by the individual laboratories agreed fairly well.

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TIME AND TEMPERATURE DURING TRANSPORT

Laboratory code no.	Date of departure	Date of arrival	Maximal temperature	Minimal temperature
A	29-8-74	3-9-74	29° C	
B	29-8-74	5-9-74	37	
C	29-8-74	5-9-74	37	
D	29-8-74	2-9-74	35	
E	29-8-74	29-8-74	26	
F	29-8-74	no data available		
G	29-8-74	29-8-74	30	
H	29-8-74	3-9-74	31	
I	29-8-74	5-9-74	30	
J	29-8-74	5-9-74	32	
K	29-8-74	3-9-74	22	
L	29-8-74	16-9-74	26	18° C
M	29-8-74	4-9-74	24	
N	29-8-74	17-9-74	-	
O	29-8-74	11-9-74	22	
P	29-8-74	3-9-74	25	18° C
Q	29-8-74	3-9-74	34	
R	29-8-74	3-9-74	24	
S	29-8-74	2-9-74	29	
T	29-8-74	17-9-74	29	
U	29-8-74	3-9-74	29	
V	29-8-74	5-9-74	21	
W	29-8-74	samples have not been analyzed		
X	29-8-74	10-9-74	-	
Y	29-8-74	4-9-74	24	
Z	29-8-74	20-9-74	21	
a	29-8-74	no data available		
b	29-8-74	26-9-74	36	14° C
c	29-8-74	samples have not arrived		
d	29-8-74	4-9-74	30	
e	29-8-74	3-9-74	21	
f	29-8-74	3-9-74	29	
g	29-8-74	3-9-74	26	16° C
h	29-8-74	29-8-74	-	

Laborat. code no.	Sample A concentration/mg/L				Sample B concentration/mg/L				Sample C concentration/mg/L						
	Date of analysis	x ₁	x ₂	x ₃	x ₄	Date of analysis	x ₁	x ₂	x ₃	x ₄	Date of analysis	x ₁	x ₂	x ₃	x ₄
A	5-9/9	0,001	0,016	0,007	0,005	9-10/9	0,005	0,010	0,004	0,008	10-11/9	2,152	2,490	2,490	1,920
C	9-17/9	1,100	1,260	1,253	0,337	19-27/9	0,337	0,553	0,506	0,025	1-15/10	26,000	28,000	20,500	
D	7/10	0,02	0,03	0,025	0,03	7/10	0,03	0,025	0,030	0,025	7/10	1,22	1,16	1,03	1,09
G	27/9	0,043	0,052	0,045	0,047	27/9	0,047	0,050	0,047	0,042	27/9	3,00	3,00	3,00	3,00
H	4-6/9	0,11	0,11	0,11	0,12	4-6/9	0,12	0,12	0,13	0,13	4-6/9	3,35	3,35	3,35	3,30
I	13/9	0,11	0,11	0,11	0,11	16/9	0,12	0,12	0,12	0,12	16/9	3,45	3,45	3,45	3,50
J	6/9	0,39	0,38	0,38	0,37	6/9	0,37	0,38	0,38	0,38	6/9	4,32	4,25	4,30	4,30
K	19/9	0,056	0,046	0,058	0,047	17/9	0,119	0,120	0,112	0,111	18/9	3,54	3,43	3,23	3,15
L	14/10	1,17	1,16		1,22	14/10	1,22	1,22		2,24	14/10	2,25	2,24		
M	10-11/9	0,142	0,136	0,139	0,153	10-11/9	0,153	0,157	0,164	0,164	10-11/9	3,42	3,40		
N	1/10	0,070	0,065		0,080	2-4/10	0,080	0,085	0,112	0,112	3/10	3,2	2,7		
P	5-11/9	0,630 0,140	0,630 0,150	0,600 0,160	0,560 0,082	5-11/9	0,560 0,082	0,570 0,085	0,530 0,097	0,530 0,097	5-11/9	3,120 5,360	3,200 6,050	3,200 5,480	3,160 5,300
Q	4-11/9	5,3 4,55 2,92	3,15		4,6 3,25 1,82	4-11/9	4,6 3,25 1,82	2,35			4-11/9	11,6 8,7 5,9	5,3		
R	5/9	2,3	2,9	2,4	1,9	5/9	3,0	3,3	2,9	3,3	5/9	6,0	5,4	6,0	5,2
S	3-22/9	0,096	0,085		n.d.	3-22/9	n.d.	n.d.	n.d.	n.d.	3-4/9	3,64	3,62		
T	19/9	0,14	0,16	0,17	0,18	19/9	0,18	0,16	0,16	0,18	19/9	3,0	3,7	3,5	3,4
U	1-2/10	0,030	0,031	0,031	0,038	1-2/10	0,038	0,035	0,038	0,033	1-2/10	3,19	3,06	3,50	3,44
V	10-27/9	0,15	0,16	0,19	0,23	18-27/9	0,23	0,20	0,23	0,23	18-27/9	2,4	3,2	3,0	2,7
X	12-13/9	0,17	0,17	0,15	0,19	12-13/9	0,19	0,20	0,18	0,14	12-13/9	3,87	3,86	3,76	
Y	30/9	0,13	0,13	0,12	0,14	30/9	0,14	0,14	0,14	0,14	1/10	4,2	4,0	3,9	4,1
Z	4/10	0,0496	0,0490	0,0497	0,050	4/10	0,023	0,0229	0,023	0,023	4/10	2,507	2,590	2,510	2,510
a	3/10	0,020	0,037	0,042	0,044	3/10	0,037	0,035	0,076	0,036	3/10	1,1	1,7	2,29	2,2
b	7-25/10	0,04	0,04	0,04	0,02	7-25/10	0,02	0,02	0,02	0,02	7-25/10	1,23	1,20	1,25	
d	5-6/9	0,150	0,165	0,160	0,150	5-6/9	0,195	0,170	0,145	0,165	5-10/9	4,875	5,00	5,125	5,125
e	4/9	0,13	0,13	0,13	0,14	4/9	0,14	0,14	0,13	0,13	4/9	2,7	2,7	2,9	
f	6-11/9	0,11	0,10	0,15	0,14	6-11/9	0,14	0,15	0,14	0,13	10-13/9	3,50	3,48	3,54	3,58
g	17/9	0,1	0,3	0,2	0,3	17/9	0,2	0,2	0,2	0,2	17/9	3,6 4,3b	3,7 4,3b	3,0	3,7
h	1/9	0,1a			0,1a	18/9	0,1a				30/8				

C Y A N I D E S - mean concentrations, lowest and highest concentrations of each laboratory

Laborat code no	Sample A			Sample B			Sample C						
	Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]	Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]	n	Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]
A	5-9/9	0,007	0,001	0,016	9-10/9	0,007	0,004	0,010	4	10-11/9	2,263	1,920	2,490
C	9-17/9	1,171	1,100	1,260	19-27/9	0,464	0,333	0,553	3	1-15/10	24,833	20,500	28,000
D	7/10	0,024	0,020	0,030	7/10	0,027	0,025	0,030	4	7/10	1,14	1,08	1,22
G	27/9	0,045	0,042	0,052	27/9	0,046	0,042	0,050	4	27/9	3,00	3,00	3,00
H	4-6/9	0,11	0,11	0,11	4-6/9	0,12	0,12	0,13	4	4-6/9	3,34	3,30	3,35
I	13/9	0,11	0,11	0,11	16/9	0,12	0,12	0,12	4	16/9	3,46	3,45	3,50
J	6/9	0,38	0,38	0,39	6/9	0,38	0,37	0,38	4	6/9	4,29	4,25	4,32
K	19/9	0,052	0,046	0,058	17/9	0,115	0,111	0,120	4	18/9	3,34	3,15	3,54
L	14/10	1,16	1,16	1,17	14/10	1,22	1,22	1,22	2	14/10	2,25	2,24	2,25
M	10-11/9	0,139	0,136	0,142	10-11/9	0,158	0,153	0,164	2	10-11/9	3,41	3,40	3,42
N	1/10	0,067	0,065	0,070	2-4/10	0,092	0,080	0,112	2	3/10	3,0	2,7	3,2
P	5-11/9	0,625	0,600	0,640	5-11/9	0,547	0,530	0,570	4	5-11/9	3,170	3,120	3,200
Q	4-11/9	0,147	0,140	0,160	4-11/9	0,090	0,082	0,097	4	4-11/9	5,548	5,300	6,050
a	5/9	4,22	3,15	5,3	4-11/9	3,5	2,35	4,6	2	4-11/9	8,7	5,8	11,6
b	3-22/9	4,55	2,3	0,096	5/9	3,25	2,9	3,3	1	5/9	8,7	5,2	6,0
c	19/9	2,92	0,085	0,18	3-22/9	1,82	n.d.	n.d.	1	3-4/9	5,6	3,62	3,64
R	1-2/10	2,4	0,14	0,18	19/9	n.d.	0,16	0,18	4	19/9	3,63	3,0	3,7
S	18-27/9	0,090	0,030	0,031	1-2/10	0,17	0,035	0,038	4	1-2/10	3,4	3,06	3,50
T	12-13/9	0,16	0,14	0,19	18-27/9	0,037	0,20	0,23	4	18-27/9	3,30	2,4	3,2
U	30/9	0,16	0,15	0,17	12-13/9	0,22	0,18	0,20	4	12-13/9	2,8	3,73	3,87
V	4/10	0,13	0,12	0,13	30/9	0,19	0,14	0,14	3	1/10	3,84	3,9	4,2
X	3/10	0,0496	0,0490	0,050	4/10	0,14	0,14	0,14	4	4/10	4,0	3,9	4,2
Y	7-25/10	0,036	0,020	0,044	3/10	0,023	0,029	0,023	4	3/10	2,529	2,507	2,590
a	5-6/9	0,04	0,04	0,04	7-25/10	0,046	0,035	0,076	4	7-25/10	1,82	1,1	2,29
b	4/9	0,156	0,150	0,165	5-10/9	0,02	0,02	0,02	3	5-10/9	1,23	1,20	1,25
d	6-11/9	0,13	0,12	0,13	4/9	0,169	0,145	0,195	4	4/9	5,031	4,875	5,125
e	18/9	0,12	0,10	0,15	6-11/9	0,14	0,13	0,14	3	6-11/9	2,8	2,7	2,9
f		0,2	0,1	0,3	18/9	0,14	0,13	0,15	4	10-13/9	3,52	3,48	3,58
g		0,1a)	0,1	0,3		0,2	0,2	0,2	4	30/8	3,5	3,0	3,7
h		0,1a)	0,1	0,3		0,1a)	0,2	0,2	1		4,3b)	4,3b)	4,3b)

P H E N O L S - individual results of measurement

Laborat. code no.	Sample A concentration (mg/l)				Sample B concentration (mg/l)				Sample C concentration (mg/l)							
	Date of analysis	x ₁	x ₂	x ₃	x ₄	Date of analysis	x ₁	x ₂	x ₃	x ₄	Date of analysis	x ₁	x ₂	x ₃	x ₄	
A	6/9	7,15	5,7	7,6	6,4	6/9	7,6	8	8,7	7,9	6/9	8,15	5,7	7,3	7,3	
B	11-13/9	11,5	12,0	11,8		11-13/9	12,4	12,0	12,4		11-13/9	11,8	12,0	11,5	7,3	
C	11-27/9	9,0	8,5	8,7		11-27/9	9,5	7,2	8,7		11-27/9	7,8	7,5	8,4	7,3	
E	6/9	7,55	7,53	7,52	7,48	6/9	7,72	7,72	7,77	7,82	9/9	8,07	8,07	8,09	7,97	
F		7,1	6,4	6,9			6,5	6,6	6,1			6,8	6,6	6,3		
G	27/9	1,19	1,19	1,19	1,19	27/9	1,38	1,40	1,36	1,40	27/9	7,75	7,85	7,55	7,35	
H	4-6/9	8,16	7,92	7,92		4-6/9	7,92	7,92	7,92		4-6/9	8,16	8,16	8,16	7,35	
I	9/9	7,44	7,48	7,44		10/9	7,60	7,56	7,46		11/9	7,62	7,44	7,68	8,16	
J	10/9	9,4	9,4	9,0	9,4	10/9	9,0	9,0	9,2	9,8	10/9	9,4	9,0	9,4	9,6	9,6
K	5/9	7,6	6,9	7,6	7,6	5-6/9	7,0	7,35	7,6	7,0	6/9	7,0	7,1	7,2	7,0	7,0
L	7/10	7,6	7,7			7/10	7,6	7,8			7/10	7,8	8,0			
II	10-11/9	7,74	7,45	7,35		10-11/9	7,40	7,39	7,47		10-11/9	7,37	7,11	7,25		
N	24-25/9	8,00	8,24	8,24		25-26/9	7,94	8,00			26-27/9	8,28	8,00	7,88		
O	19/9	8,7	8,7			19/9	8,7	8,7			19/9	9,2	9,25			
P	4/9	2,86	2,88	2,90	2,80	4/9	2,96	2,98	2,83	2,97	4/9	2,80	2,70	2,70	3,00	
Q	4-9/9	8,00	8,40	8,30	8,00	4-9/9	7,80	8,80	7,90	7,80	4-9/9	7,60	8,50	8,50	8,00	8,00
R	5/9	7,35	7,65	7,60	7,40	5/9	7,60	8,00	8,00	7,60	5/9	7,70	7,80	7,80	7,80	7,80
S	3-5/9	6,93	6,68			3-5/9	7,05	6,84			3-5/9	7,07	7,00			
T	23/9	8,8	9,2	9,1		23/9	8,4	8,8	9,0		23/9	8,8	8,7	8,8		
U	16-17/9	7,45	7,75	7,47	7,50	16-17/9	7,50	7,83	7,75	7,87	16-17/9	8,20	8,13	8,30	8,10	
V	18-27/9	7,6	7,6	7,7	7,6	18-27/9	6,6	6,6	6,6	6,6	18-27/9	6,9	7,0	6,9	6,9	6,9
W	12-13/9	16,0	13,7	12,7	7,40	12-13/9	14,8	12,2	16,4	7,60	5/9	15,8	14,1	15,7	7,3	
X	30/9	7,6	7,7	7,8	7,6	1/10	7,7	7,7	7,6	7,7	1/10	7,8	7,7	7,7	7,7	7,7
Z	4/10	7,57	7,51	7,85	7,85	4/10	7,97	8,24	7,60	7,66	4/10	7,73	7,62	8,15	8,18	8,18
a	3/10	6,0	6,1	5,35	5,25	3/10	7,7	7,75	7,0	7,2	3/10	7,5	7,25	7,3	7,3	7,3
b	7-25/10	7,85	7,85	7,85	8,00	7-25/10	7,85	7,85	7,65	8,15	7-25/10	7,85	13,03	8,09	8,09	8,09
d	6/9	8,00	8,00	8,00	8,00	6/9	7,95	8,10	8,00	8,15	6/9	7,95	7,85	8,00	8,00	8,00
e	4/9	9,5	9,6	9,3	6,2	4/9	6,8	6,9	6,6	6,6	4/9	9,1	9,1	9,2	9,2	9,2
f	11-16/9	6,6	6,1	6,5	6,2	13-16/9	6,4	6,4	6,7	6,6	13-16/9	6,4	6,2	6,3	6,2	6,2
g		3,0					2,5					0,25				
h	30/8	7,1	7,1	6,6	6,6	30/8	7,5	7,4	7,3	6,3	30/8	7,5	7,5	6,8	6,8	7,3

P.H.E.N.O.L.S - mean concentrations, lowest and highest concentrations of each laboratory

Laborat. code no.	Date of arrival	Max. Temp. [°C]	S a m p l e A			S a m p l e B			S a m p l e C							
			Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]	Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]	Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]		
A	3/9	28,5	6,71	5,7	7,6	8,05	7,6	8,7	6/9	8,05	7,6	8,7	6/9	7,1	5,7	8,15
B	5/9	37	11,8	11,5	12,0	12,3	12,0	12,4	11-13/9	12,3	12,0	12,4	11-13/9	11,8	11,5	12,0
C	5/9	37	8,7	8,5	9,0	8,5	7,2	9,5	11-27/9	8,5	7,2	9,5	11-27/9	7,9	7,5	8,4
E	29/8	26	7,52	7,48	7,55	7,76	7,72	7,82	6/9	7,76	7,72	7,82	9/9	8,05	7,97	8,09
F			6,8	6,4	7,1	6,4	6,1	6,6		6,4	6,1	6,6		6,6	6,3	6,8
G	29/8	30	1,19	1,19	1,19	1,39	1,36	1,40	27/9	1,39	1,36	1,40	27/9	7,83	7,75	7,95
H	2/9	30	8,00	7,92	8,16	7,86	7,68	7,92	4-6/9	7,86	7,68	7,92	4-6/9	8,16	8,16	8,16
I	5/9	26	7,45	7,44	7,48	7,54	7,46	7,60	9/9	7,54	7,46	7,60	9/9	7,58	7,44	7,68
J	5/9	32	9,3	9,0	9,4	9,3	9,0	9,8	10/9	9,3	9,0	9,8	10/9	9,4	9,0	9,6
a)			9,4	9,4	9,5	9,7	9,6	10,0		9,7	9,6	10,0		9,5	9,4	9,6
b)			7,4	6,9	7,6	7,24	7,0	7,6	5-6/9	7,24	7,0	7,6	6/9	7,08	7,0	7,2
K	3/9	28	7,6	7,6	7,7	7,7	7,6	7,8	7/10	7,7	7,6	7,8	7/10	7,9	7,8	8,0
L	16/9	26	7,51	7,35	7,74	7,42	7,39	7,47	10-11/9	7,42	7,39	7,47	10-11/9	7,24	7,11	7,37
M	4/9	24	8,16	8,00	8,24	7,97	7,94	8,00	25-26/9	7,97	7,94	8,00	26-27/9	8,05	7,88	8,28
N	17/9															
O	11/9	22	8,7	8,7	8,7	8,7	8,7	8,7	19/9	8,7	8,7	8,7	19/9	9,2	9,2	9,25
P	3/9	25	2,86	2,80	2,90	2,94	2,83	2,98	4/9	2,94	2,83	2,98	4/9	2,80	2,70	3,00
Q	3/9	34	8,17	8,00	8,40	8,07	7,80	8,80	4-9/9	8,07	7,80	8,80	4-9/9	8,15	7,60	8,50
R	3/9	24	7,50	7,35	7,65	7,80	7,60	8,00	5/9	7,80	7,60	8,00	5/9	7,80	7,70	7,80
S	2/9	29	6,80	6,68	6,93	6,95	6,84	7,05	3-5/9	6,95	6,84	7,05	3-5/9	7,04	7,00	7,07
T	17/9	29	9,0	8,8	9,2	8,7	8,4	9,0	23/9	8,7	8,4	9,0	23/9	8,8	8,7	8,8
U	3/9	29	7,54	7,45	7,75	7,74	7,50	7,87	16-17/9	7,74	7,50	7,87	16-17/9	8,18	8,10	8,30
V	5/9	21	7,6	7,6	7,7	6,6	6,6	6,6	18-27/9	6,6	6,6	6,6	18-27/9	6,9	6,9	7,0
a)			7,8	7,8	7,8	6,8	6,8	6,8		6,8	6,8	6,8		7,0	7,0	7,0
b)			14,1	12,7	16,0	14,5	12,2	16,4	12-13/9	14,5	12,2	16,4	12-13/9	15,2	14,1	15,8
X	10/9								1/10	7,7	7,6	7,7	1/10	7,7	7,7	7,8
Y	4/9	24	7,7	7,6	7,8	7,87	7,60	8,24	4/10	7,87	7,60	8,24	4/10	7,92	7,62	8,18
Z	20/9	21	7,69	7,51	7,85	7,53	7,0	7,75	3/10	7,53	7,0	7,75	3/10	7,35	7,25	7,5
a			5,54	5,25	6,1	5,78	5,65	6,1	7-25/10	5,78	5,65	6,1	7-25/10	9,66	7,85	13,03
b	26/9	36	7,85	7,85	7,85	8,05	7,95	8,15	6/9	8,05	7,95	8,15	6/9	7,95	7,85	8,00
d	4/9	30	8,00	8,00	8,00	6,8	6,6	6,9	4/9	6,8	6,6	6,9	4/9	9,1	9,1	9,2
e	3/9	21	9,5	9,3	9,6	6,5	6,4	6,7	13-16/9	6,5	6,4	6,7	13-16/9	6,3	6,2	6,4
f	3/9	29	6,4	6,1	6,6	2,5	2,5	2,5		2,5	2,5	2,5		0,25	0,25	0,25
g	3/9	26	3,0	3,0	3,0	7,1	6,3	7,5	30/8	7,1	6,3	7,5	30/8	7,4	6,8	7,5
h	30/8	20	6,9	6,6	7,1											

TOTAL ORGANIC CARBON - individual results of measurement

Laborat. code no.	Date of analysis	Sample A concentration (mg/L)				Date of analysis	Sample B concentration (mg/L)				Date of analysis	Sample C concentration (mg/L)			
		x ₁	x ₂	x ₃	x ₄		x ₁	x ₂	x ₃	x ₄		x ₁	x ₂	x ₃	x ₄
A a)	24/9	44	43,5	45	43,5	24/9	41,5	40	41	43	24/9	49	49,5	52	49,5
A b)		53	53	54	52		45	46,5	48,5	47,5		55	56,5	54	55
U a)	1/10	47	43,5	43	40,5	1/10	41	41,5	40,5	41	1/10	53	51,5	48,5	51
U b)		46,5	49,5	49	49		43	45,5	45	45		61	56,5	56	56
Y	30/9	52	52	52	52	30/9	46	46	46	45	30/9	55	55	55	55
e	4/9	49	47			4/9	47	44			4/9	51	49		
f	9/9	46,8	45,2	46,8		9/9	46,0	50,0	48,4	45,2	9/9	51,6	50,0	55,2	51,6
g		40,3					39,8					50,5			

ALIPHATIC HYDROCARBONS - individual results of measurement

Laborat. code no.	Date of analysis	Sample A concentration (mg/L)				Date of analysis	Sample B concentration (mg/L)				Date of analysis	Sample C concentration (mg/L)			
		x ₁	x ₂	x ₃	x ₄		x ₁	x ₂	x ₃	x ₄		x ₁	x ₂	x ₃	x ₄
B	11-13/9	<0,4	<0,4	<0,4	<0,4	11-13/9	<0,4	<0,4	<0,4	<0,4	11-13/9	<0,4	<0,4	<0,4	<0,4
C	16-18/9	0,8	1,2	0,9	<0,4	16-18/9	<0,4	<0,4	<0,4	<0,4	16-18/9	<0,4	<0,4	<0,4	<0,4
E	14/10	0,5	0,5	0,5	0,5	14/10	0,7	0,9	0,8	0,8	14/10	0,9	0,13	0,17	0,17
F		1,18					0,41					0,42			
G	27/9	3,8	2,1	4,4	4,4	27/9	1,1	1,8	1,6	2,1	27/9	3,5	3,2	2,8	2,9
H	10-17/9	2,45	2,28	2,38	2,52	10-12/9	0,70	0,68	0,49	0,52	10-12/9	0,24	0,52	0,35	0,32
K	13/9	4,1	3,9	3,9	4,2	12/9	1,8	1,8	1,6	1,3	11/9	1,2	1,2	1,3	1,4
J		3	2,4	2,2	1,8		1,5	1,1	1,1	0,8		1,6	0,9	1,2	0,7
M	24/9	14,2	11,3			24/9	12,2	10,9			24/9	11,4	4,73		
N a)	1/10	0,5	0,6			1/10	0,4	1,0			1/10	3	3,6	3,7	4,0
N b)		3,4	6,2	6,7	>10		2	2,4	2,8	3,5		0,4	0,4		
O	20/9	1,75	1,7			20/9	0,4	0,4			20/9	0,4	0,4		
Q a)	13/9	0,5	0,5			13/9	0,25	0,25			13/9	0,17	0,17		
Q b)		2,9	2,9				1,3	1,3				1,2	1,2		
S	4/9	1,8	1,8	1,8	1,8	4/9	0,4	0,4	0,4	0,4	4/9	0,4	0,4	0,4	0,4
V	18-27/9	<2				18-27/9	<2				18-27/9	<2			
f	18/9	7,4	3,5			18/9	7,0	2,0			18/9	2,4	2,0		
h	2/9	0,7	0,7	0,5	0,5	2/9	0,5	0,4			2/9	0,1	0,1	0,1	0,1

TOTAL ORGANIC CARBON - mean concentrations, lowest and highest concentrations of each laboratory

Laborat. code no.	Date of arrival	Max. Temp. (°C)	Sample A			Sample B			Sample C					
			Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]	Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]	Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]
A	3/9	28,5	24/9	44	43,5	45	24/9	41,5	40	43	24/9	50	49	52
				n	53	54		n	47	45		n	55	54
U	3/9	29	1/10	44	40,5	47	1/10	41	40,5	41,5	1/10	51	49,5	53
				n	48,5	46,5		49,5	n	44,5		43	45,5	n
Y	4/9	24	30/9	52	52	52	30/9	46	45	46	30/9	55	55	55
				n	48	47		49	n	46		44	47	n
f	3/9	26	9/9	46,3	45,2	46,8	9/9	47,4	45,2	50,0	9/9	52,1	50,0	55,2
				n	40,3			1	39,8			1	50,8	

ALIPHATIC HYDROCARBONS - mean concentrations, lowest and highest concentrations of each laboratory

Laborat. code no.	Date of arrival	Max. Temp. (°C)	Sample A			Sample B			Sample C					
			Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]	Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]	Date of analysis	\bar{x} [mg/l]	x_{min} [mg/l]	x_{max} [mg/l]
B	5/9	37	11-13/9	<0,4	<0,4	<0,4	11-13/9	<0,4	<0,4	<0,4	11-13/9	<0,4	<0,4	<0,4
				n	1,0	0,8		1,2	n	<0,4		<0,4	<0,4	n
C	5/9	37	16-13/9	0,5	0,5	0,5	16-18/9	0,8	0,7	0,9	16-18/9	0,34	0,13	0,9
				n	1,18				n	0,41				n
E	29/8	26	14/10	3,7	2,1	4,4	27/9	1,7	1,1	2,1	27/9	3,1	2,8	3,5
				n	2,41	2,28		2,52	n	0,60		0,49	0,70	n
G	2/9	30	10-12/9	4,0	3,9	4,2	10-12/9	1,6	1,3	1,8	10-12/9	1,3	1,2	1,4
				n	2,4	1,8		3	n	1,1		0,8	1,5	n
H	3/9	28	13/9	12,8	11,3	14,2	12/9	11,6	10,9	12,2	11/9	8,06	4,73	11,4
				n	0,6	0,5		0,6	n	0,7		0,4	1,0	n
I	16/9	26	24/9	4,4	3,4	>10	24/9	2,7	2	3,5	24/9	3,6	3	4,0
				n	1,7	1,7		1,75	n	0,4		0,4	0,4	n
N	17/9	22	1/10	0,5	0,5	0,6	1/10	0,25	0,4	0,4	1/10	0,17	0,17	0,17
				n	4,4	3,4		10	n	2,7		2	3,5	n
O	11/9	34	20/9	1,7	1,7	1,75	20/9	0,4	0,4	0,4	20/9	0,4	0,4	0,4
				n	0,5	0,5		0,5	n	0,25		0,25	0,25	n
Q	3/9	29	13/9	2,8	2,9	2,9	13/9	1,3	1,3	1,3	13/9	1,2	1,2	1,2
				n	1,8	1,8		1,8	n	0,4		0,4	0,4	n
S	2/9	29	4/9	<2	<2	<2	4/9	<2	<2	<2	4/9	<2	<2	<2
				n	5,5	3,5		7,4	n	4,5		2,0	7,0	n
V	5/9	29	13-27/9	0,6	0,5	0,7	18-27/9	0,5	0,4	0,5	18-27/9	0,1	0,1	0,1
				n	5,5	3,5		7,4	n	4,5		2,0	7,0	n
f	3/9	29	2/9	0,6	0,5	0,7	2/9	0,5	0,4	0,5	2/9	0,1	0,1	0,1
				n	5,5	3,5		7,4	n	4,5		2,0	7,0	n

Determination of C Y A N I D E S

Analytical methods used by the participating laboratories

Laboratory code no.	Sample pretreatment	Analytical determination	Concentration range* /mg/l	Detection limit /mg/l
A	distillation	colorimetric (pyridine-pyrazolone at 620 nm)	<0,5	0,001
C	distillation	colorimetric (pyridine-pyrazolone at 620 nm)	0,002-0,1	0,002
D	-	ionselective electrode (standard addition)	0,01-100	0,01
C	-	colorimetric (pyridine-barbituric acid at 580 nm)		0,005
H	distillation	colorimetric (pyridine-pyrazolone at 620 nm)	1 - 10	0,01
I	distillation	colorimetric (pyridine-pyrazolone at 620 nm)	1 - 10	0,01
J	neutralisation	colorimetric (pyridine-pyrazolone at 620 nm)	0,01 - 0,1	0,01
K	distillation	colorimetric (pyridine-pyrazolone at 620 nm)	0 - 0,1	0,001
L	distillation	colorimetric (pyridine-pyrazolone)		
M	distillation	colorimetric (pyridine-pyrazolone at 620 nm)	0,01 - 1	0,01
N	distillation	colorimetric (pyridine-pyrazolone)		
P	a) without distillation b) with distillation	colorimetric (pyridine-pyrazolone at 620 nm)	<0,05	0,01
Q	distillation	a) ionselective electrode b) titrimetric (p-dimethylaminobenzalrhodanide) c) colorimetric (pyridine-pyrazolone at 620 nm)		
R	distillation	ionselective electrode		0,05
S	distillation	colorimetric (pyridine-pyrazolone at 620 nm)		
T	-	ionselective electrode		
U	distillation	colorimetric (pyridine-p-phenylenediamine at 515 nm)	0,05 - 250	0,05
V	distillation	colorimetric (pyridine-p-phenylenediamine at 515 nm)	0,02 - 1	0,01
X	distillation at pH 7	colorimetric (pyridine-p-phenylenediamine at 515 nm)	< 1	0,02
Y	distillation	colorimetric (pyridine-p-phenylenediamine at 515 nm)		0,01
Z	distillation	colorimetric (pyridine-pyrazolone at 620 nm)	0,01 - 1	0,02
a	distillation	colorimetric (pyridine-pyrazolone at 620 nm)	0,01 - 1	0,001
b	distillation	colorimetric (pyridine-pyrazolone at 620 nm)	0,01 - 1	0,001
d	distillation	colorimetric (pyridine-barbituric acid at 580 nm)	0,01 - 1	0,001
e	distillation	colorimetric (pyridine-pyrazolone at 630 nm)	0,01 - 1	0,001
f	distillation	colorimetric (pyridine-pyrazolone at 620 nm)	0,01 - 1	0,001
g	distillation	titrimetric (p-dimethylaminobenzalrhodanide)	0,02 - 0,5	0,02
h	distillation	a) colorimetric b) titrimetric (by Tyndall-effect)	0 - 10 4 - 20 >0,1	0,005 2 0,1

*) the method is normally used in for analysis of water samples

Determination of P H E N O L S
Analytical methods used by the participating laboratories

Laboratory code no.	Sample pretreatment	Analytical determination	Concentration range* mg/L	Detection limit mg/L
A	filtration	colorimetric (4-amino-antipyrine at 530 nm), CHCl ₃ -extraction	<1,5	0,005
B	extraction (ether at pH2)	colorimetric (diazotized sulfanilic acid at 445 nm)		1
C	extraction (ether at pH2)	colorimetric (p-nitraniline at 475 nm)		0,01
E	filtration, neutralisation	colorimetric (4-amino-antipyrine at 470 nm), CHCl ₃ -extraction	>0,2 river water, ground water	0,005
F	extraction (di-ise-propyl-ether at pH 3)	colorimetric (4-amino-antipyrine at 460 nm), CHCl ₃ -extraction	0,0005-0,01	0,0005
G	-	colorimetric (4-amino-antipyrine)	0,2 - 2	0,1
H	distillation	colorimetric (4-amino-antipyrine at 510 nm)	1 - 50	0,1
I	distillation	colorimetric (4-amino-antipyrine at 460 nm), CHCl ₃ -extraction	0 - 1	0,005
J	distillation	a) colorimetric (4-amino-antipyrine at 510 nm) b) colorimetric (p-nitraniline at 472 nm)	1	0,1
K	distillation	colorimetric (4-amino-antipyrine at 460 nm), CHCl ₃ -extraction	0 - 2	0,01
L	distillation	colorimetric (4-amino-antipyrine)		
M	distillation	colorimetric (4-amino-antipyrine), CHCl ₃ -extraction		
N	filtration, distillation	colorimetric (4-amino-antipyrine)		
O	-			
P	distillation	colorimetric (4-amino-antipyrine at 510 nm)	0,02 - 10	0,02
Q	distillation	colorimetric (4-amino-antipyrine at 460 nm), CHCl ₃ -extraction	0,01 - 1	0,02
R	homogenisation, distillation	colorimetric (4-amino-antipyrine at 510 nm)	>0,5	0,02
S	distillation	colorimetric (4-amino-antipyrine at 510 nm)		
T	-	colorimetric (4-amino-antipyrine at 510 nm)	1 - 5	0,05
U	distillation	colorimetric (4-amino-antipyrine at 460 nm), CHCl ₃ -extraction	0,01 - 1	0,01
V	a) neutralisation b) distillation	a) GIG for phenol and cresol b) colorimetric (4-amino-antipyrine at 510 nm)	<0,1	b) 0,1
X	distillation	colorimetric (4-amino-antipyrine at 460 nm), CHCl ₃ -extraction		0,005
Y	distillation	colorimetric (4-amino-antipyrine at 510 nm)	0 - 50	0,1
Z	distillation	colorimetric (4-amino-antipyrine at 510 nm)	0 - 50	1
a	distillation	colorimetric (4-amino-antipyrine at 510 nm), CHCl ₃ -extraction	0,5 - 5 surface water, drinking water	0,0005
b	distillation	colorimetric (4-amino-antipyrine at 510 nm)	0,5 - 5 surface water	0,0005
d	distillation	colorimetric (4-amino-antipyrine at 510 nm)	0,2 - 10	0,2
e	-	colorimetric (4-amino-antipyrine at 465 nm), CHCl ₃ -extraction	0 - 0,2 surface water 0 - 10 effluent	0,01
f	distillation	colorimetric (4-amino-antipyrine at 460 nm), CHCl ₃ -extraction	0 - 1	1
g	extraction (ether)	TIC for phenol, cresol and Xylenol	drinking water	
h	distillation	colorimetric (uranylacetate-resagent)		

*) the method is normally used in for analysis of water samples

Determination of TOTAL ORGANIC CARBON
Analytical methods used by the participating laboratories

Laboratory code no.	Sample pretreatment	Analytical determination	Principle of the determination	Concentration range*) [mg/l]	Detection limit [mg/l]
A	a) unmixed probe b) mixed probe	Beckman TOC-analyser with IR-detector; standard: K-bipthalate, subtraction of inorganic carbon	$C_{org} \rightarrow CO_2$	<100	5
U	a) centrifugated probe b) mixed probe acidification with HNO_3 (pH 3) and degasing of CO_2	Phasesep TOCSIN-analyser with flame-ionisation-detector	$C_{org} \rightarrow CO_2 \rightarrow CH_4$	0,1 - 1000	0,1 - 5,5
Y	acidification with HNO_3 (pH 3) and degasing of CO_2	Phasesep TOCSIN-analyser with flame-ionisation-detector	$C_{org} \rightarrow CO_2 \rightarrow CH_4$		
e	acidification with HNO_3 (pH 3,5-4) and degasing of CO_2	Beckman TOC-analyser with IR-detector; standard: K-bipthalate	$C_{org} \rightarrow CO_2$	0 - 25 surface water	1
f	filtrated probe acidification (pH 2 - 3) and degasing of CO_2	Beckman TOC-analyser with IR-detector	$C_{org} \rightarrow CO_2$	0 - 20	1
g	acidification with HNO_3 and degasing of CO_2	Phasesep TOCSIN-analyser with flame-ionisation-detector	$C_{org} \rightarrow CO_2 \rightarrow CH_4$		0,1

*) the method is normally used in for analysis of water samples

Determination of H Y D R C A R B O N S
Analytical methods used by the participating laboratories

Laborat. code no.	Sample pretreatment	Precleaning	Analytical determination	Concentration range*) /mg/l/	Detection limit /mg/l/
B	homogenisation, acidification, extraction	Florisil (column)	IR at 2870/2925/2960 cm ⁻¹ ; 1 cm quartz-cells standard: iso-octane/hexadecane/benzene	0,1 - 8 river-, groundwater	0,4
C	homogenisation, acidification, extraction	Florisil (column)	IR at 2870/2925/2960 cm ⁻¹ ; 1 cm quartz-cells standard: iso-octane/hexadecane/benzene	0,1 - 8 river-, groundwater	0,4
E	extraction	Al ₂ O ₃ (column)	IR at 2870/2925/2960 cm ⁻¹ ; 5 cm quartz-cells standard: gasoil	0,1 - 8 river-, groundwater	0,05
F	extraction	Florisil (batch)	IR at 2870/2925/2960 cm ⁻¹ ; 5 cm quartz-cells standard: API-mixture	0,05-0,5 river-, groundwater	0,05
G	extraction	-	IR at 2870/2925/2960 cm ⁻¹ ; 5 cm quartz-cells standard: gasoil		0,05
H	acidification (pH 5), extraction	Florisil (batch)	IR at 2870/2925 cm ⁻¹ ; 4 cm quartz-cells standard: hexadecane	0,01 - 10	0,01
K	acidification (pH 5), extraction	-	IR at 2870 cm ⁻¹ ; 4 cm quartz-cells standard: iso-octane	0 - 25	0,2
L	extraction; use of Na ₂ SO ₄ anhydr.	Florisil (column)	IR; standard: benzene/cetane/trimethylpentane		
M	acidification (pH 1), extraction	-	IR; standard: API-mixture		
N	extraction a) with and b) without centrifugation	-	IR		
O	extraction	-	IR		
Q	acidification, extraction, use of Na ₂ SO ₄ anhydr.	a) with and b) without Florisil (column)	IR at 2925 cm ⁻¹ ; 4 cm quartz-cells standard: hexadecane	>0,1	0,1
S	acidification (pH 5), extraction, use of Na ₂ SO ₄ anhydr.	Florisil (column)	IR at 2925 cm ⁻¹		0,2
V	acidification, extraction	Florisil (column)	IR: 2800 to 3100 cm ⁻¹ ; 0,1 mm KBr-cells		2
f	acidification, extraction (petrol ether), use of Na ₂ SO ₄ anhydr.	-	Evaporation of the extract and gravimetric determination of the residue	1	1
h	extraction	-	IR		

*) the method is normally used in for analysis of water samples

LAB	INTERLABORATORY TEST				MEAN	ST.DEV.
	SAMPLE A					
	MEASURED CONCENTRATION (MICROGRAM/LITER)					
H	110	110	110	110	110.0	
K	56	46	58	47	51.8	6.13
R	2300*	2900*	2400*	1900*	2375.0	411.30
T	140	160	170	180	162.5	17.08
FF	110	100	150	110	117.5	22.17
G	43	52	45	42	45.5	4.51
Y	130	130	120	130	127.5	5.00
DD	150	165	160	150	156.3	7.50
S	96	85			90.5	7.78
I	110	110	110	110	110.0	
EE	130	130	130	120	127.5	5.00
U	30	31	31	30	30.5	0.58
Q	5300*	4550*	3150*	2920*	3980.0	1137.22
J	390*	380*	380*	380*	382.5	5.00
V	150	160	190	140	160.0	21.60
N	70	65			67.5	3.54
A	1	16	7	5	7.3	6.34
L	1170*	1160*			1165.0	7.07
C	1100*	1260*	1153*		1171.0	81.50
GG	100	300*	300*	200	225.0	95.74
D	20	30	25	20	23.8	4.79
X	170	170	150		163.3	11.55
P1	140	150	160	140	147.5	9.57
P2	630*	630*	600*		620.0	17.32
M	142	136	139		139.0	3.00
Z	50	50	50	50	50.0	
AA	20	37	42	44	35.8	10.90
BB	40	40	40		40.0	
HH	100					

MEAN OF 80 MEASUREMENTS = 95.0 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)

53.7 MICROGRAM/LITER = 56.5 PERCENT

STANDARD DEVIATION (MEAN)

6.0 MICROGRAM/LITER = 6.3 PERCENT

CONFIDENCE INTERVAL (95%)

12.1 MICROGRAM/LITER = 12.7 PERCENT

NOTE:

DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS.
 NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS .
 USED BY A LABORATORY.

8.9.

INTERLABORATORY TEST		SAMPLE B			CYANID	
LAB	MEASURED CONCENTRATION (MICROGRAM/LITER)				MEAN	ST.DEV.
H	120	120	130	130	125.0	5.77
K	119	120	112	111	115.5	4.65
R	3000*	3300*	2900*	3300*	3125.0	206.16
T	180	160	160	180	170.0	11.55
FF	140	150	140	130	140.0	8.16
G	47	50	47	42	46.5	3.32
Y	140	140	140	140	140.0	
DD	195	170	145	165	168.8	20.56
S	NN	NN	NN	NN		
I	120	120	120	120	120.0	
EE	140	140	130		136.7	5.77
U	38	35	38	38	37.3	1.50
Q	4600*	3250*	2350*	1820*	3005.0	1216.18
J	370*	380*	380*	380*	377.5	5.00
V	230	200	230	230	222.5	15.00
N	80	85	112		92.3	17.21
A	5	10	4	8	6.8	2.75
L	1220*	1220*			1220.0	
C	333*	553*	506*		464.0	115.86
GG	200	200	200	200	200.0	
D	30	25	30	25	27.5	2.89
X	190	200	180		190.0	10.00
P1	82	85	97	97	90.3	7.89
P2	560*	570*	530*	530*	547.5	20.62
M	153	157	164		158.0	5.57
Z	23	23	23	23	23.0	
AA	37	35	76	36	46.0	20.02
BB	20	20	20		20.0	
HH	100					

MEAN OF 80 MEASUREMENTS = 107.6 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)

65.0 MICROGRAM/LITER = 60.4 PERCENT

STANDARD DEVIATION (MEAN)

7.3 MICROGRAM/LITER = 6.8 PERCENT

CONFIDENCE INTERVAL (95%)

14.6 MICROGRAM/LITER = 13.6 PERCENT

NOTE:

DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS.
NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS
USED BY A LABORATORY.

LAB	INTERLABORATORY TEST				MEAN	ST.DEV.
	SAMPLE C					
	MEASURED CONCENTRATION (MICROGRAM/LITER)					
H	3350	3350	3350	3330	3345.0	10.00
K	3540	3430	3230	3150	3337.5	179.14
R	6000	5400	6000	5200	5650.0	412.31
T	3000	3700	3500	3400	3400.0	294.39
FF	3500	3480	3540	3580	3525.0	44.35
G	3000	3000	3000	3000	3000.0	
Y	4200	4000	3900	4100	4050.0	129.10
DD	4875	5000	5125	5125	5031.3	119.68
S	3640	3620			3630.0	14.14
I	3450	3450	3450	3500	3462.5	25.00
EE	2700	2700	2900		2766.7	115.47
U	3190	3060	3500	3440	3297.5	207.59
Q	11600*	8700*	5800	5900	8000.0	2750.76
J	4320	4250	4300	4300	4292.5	29.86
V	2400	3200	3000	2700	2825.0	350.00
N	3200	2700			2950.0	353.55
A	2152	2490	2490	1920	2263.0	278.70
L	2250	2240			2245.0	7.07
C	26000*	28000*	20500*		24833.3	3883.73
GG	3600	3700	3000	3700	3500.0	336.65
D	1220	1160	1080	1090	1137.5	65.51
X	3870	3860	3780		3836.7	49.33
P1	5360	6050	5480	5300	5547.5	343.26
P2	3120	3200	3200	3160	3170.0	38.30
M	3420	3400			3410.0	14.14
Z	2507	2590	2510	2510	2529.3	40.52
AA	1100	1700	2290	2200	1822.5	547.14
BB	1230	1200	1250		1226.7	25.17
HH	4300	4300			4300.0	

MEAN OF 97 MEASUREMENTS = 3391.8 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)

1170.1 MICROGRAM/LITER = 34.5 PERCENT

STANDARD DEVIATION (MEAN)

118.8 MICROGRAM/LITER = 3.5 PERCENT

CONFIDENCE INTERVAL (95%)

238.7 MICROGRAM/LITER = 7.0 PERCENT

NOTE:

DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS.
 NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS
 USED BY A LABORATORY.

INTERLABORATORY TEST		SAMPLE A			PHENDL	
LAB	MEASURED CONCENTRATION (MICROGRAM/LITER)			MEAN	ST.DEV.	
H	8160	7920	7920		8000.0	138.56
K	7600	6900	7600	7600	7425.0	350.00
R	7350	7650	7600	7400	7500.0	147.20
F	7100	6400	6900		6800.0	360.56
T	8800	9200	9100		9033.3	208.17
FF	6600	6100	6500	6200	6350.0	238.05
G	1190*	1190*	1190*	1190*	1190.0	
Y	7600	7700	7800	7600	7675.0	95.74
D	8700	8700			8700.0	
DD	8000	8000	8000	8000	8000.0	
S	6830	6680			6755.0	106.07
I	7440	7480	7440		7453.3	23.09
EE	9500	9600	9300		9466.7	152.75
U	7450	7750	7470	7500	7542.5	139.85
B	11500*	12000*	11800*		11766.7	251.66
Q	8000	8400	8300	8000	8175.0	206.16
J1	9400	9400	9000	9400	9300.0	200.00
J2	9400	9500	9400	9500	9450.0	57.74
V1	7600	7600	7700	7600	7625.0	50.00
V2	7800	7800			7800.0	
N	8000	8240	8240		8160.0	138.56
A	7150	5700	7600	6400	6712.5	837.03
L	7600	7700			7650.0	70.71
E	7550	7530	7520	7480	7520.0	29.44
C	9000	8500	8700		8733.3	251.66
GG	3000*					
P	2860*	2880*	2900*	2800*	2860.0	43.20
X	16000*	13700*	12700*		14133.3	1692.14
M	7740	7745	7350		7611.7	226.62
Z	7570	7510	7850	7850	7695.0	180.65
AA	6000	6100	5350*	5250*	5675.0	436.84
BB	7850	7850	7850		7850.0	
HH	7100	7100	6600		6933.3	288.68

MEAN OF 92 MEASUREMENTS = 7803.4 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)

884.8 MICROGRAM/LITER = 11.3 PERCENT

STANDARD DEVIATION (MEAN)

92.3 MICROGRAM/LITER = 1.2 PERCENT

CONFIDENCE INTERVAL (95%)

185.3 MICROGRAM/LITER = 2.4 PERCENT

NOTE:

DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS. NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS USED BY A LABORATORY.

INTERLABORATORY TEST		SAMPLE 8			PHENOL	
LAB	MEASURED CONCENTRATION (MICROGRAM/LITER)				MEAN	ST.DEV.
H	7920	7920	7920	7680	7860.0	120.00
K	7000	7350	7600	7000	7237.5	292.62
R	7600	8000	8000	7600	7800.0	230.94
F	6500	6600	6100		6400.0	264.58
T	8400	8800	9000		8733.3	305.51
FF	6400	6400	6700	6600	6525.0	150.00
G	1380*	1400*	1360*	1400*	1385.0	19.15
Y	7700	7700	7600	7700	7675.0	50.00
O	8700	8700			8700.0	
DD	7950	8100	8000	8150	8050.0	91.29
S	7050	6840			6945.0	148.49
I	7600	7560	7460		7540.0	72.11
EE	6800	6900	6600		6766.7	152.75
U	7500	7830	7750	7870	7737.5	166.01
B	12400*	12000*	12400*		12266.7	230.94
Q	7800	8800	7900	7800	8075.0	485.63
J1	9000	9000	9200	9800*	9250.0	378.59
J2	10000*	9600	9600	9600	9700.0	200.00
V1	6600	6600	6600	6600	6600.0	
V2	6800	6800			6800.0	
N	7940	8000			7970.0	42.43
A	7600	8000	8700	7900	8050.0	465.47
L	7600	7800			7700.0	141.42
E	7720	7720	7770	7820	7757.5	47.87
C	9500	7200	8700		8466.7	1167.62
GG	2500*					
P	2960*	2980*	2830*	2970*	2935.0	70.47
X	14800*	12200*	16400*		14466.7	2119.75
M	7400	7390	7470		7420.0	43.59
Z	7970	8240	7600	7660	7867.5	296.58
AA	7700	7750	7000	7200	7412.5	370.53
BB	7500	7400	7300	6300	7125.0	556.03
HH	7100	7100	6600		6933.3	288.68

MEAN OF 93 MEASUREMENTS = 7657.0 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)
790.9 MICROGRAM/LITER = 10.3 PERCENT

STANDARD DEVIATION (MEAN)
82.0 MICROGRAM/LITER = 1.1 PERCENT

CONFIDENCE INTERVAL (95%)
164.8 MICROGRAM/LITER = 2.2 PERCENT

NOTE:
DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS.
NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS
USED BY A LABORATORY.

INTERLABORATORY TEST		SAMPLE C			PHENOL	
LAB	MEASURED CONCENTRATION (MICROGRAM/LITER)				MEAN	ST.DEV.
H	8160	8160	8160	8160	8160.0	
K	7000	7100	7200	7000	7075.0	95.74
R	7700	7800	7800	7800	7775.0	50.00
F	6800	6600	6300		6566.7	251.66
T	8800	8700	8800		8766.7	57.74
FF	6400	6200	6300	6200	6275.0	95.74
G	7750	7850	7850	7850	7825.0	50.00
Y	7800	7700	7700	7700	7725.0	50.00
O	9200	9250			9225.0	35.36
DD	7950	7850	8000	8000	7950.0	70.71
S	7070	7000			7035.0	49.50
I	7620	7440	7680		7580.0	124.90
EE	9100	9100	9200		9133.3	57.74
U	8200	8130	8300	8100	8182.5	88.84
B	11800*	12000*	11500*		11766.7	251.66
Q	7600	8500	8500	8000	8150.0	435.89
J1	9400	9000	9400	9600	9350.0	251.66
J2	9500	9600	9500	9400	9500.0	81.65
V1	6900	7000	6900	6900	6925.0	50.00
V2	7000	7000			7000.0	
N	8280	8000	7880		8053.3	205.26
A	8150	5700*	7300	7300	7112.5	1023.37
L	7800	8000			7900.0	141.42
E	8070	8070	8090	7970	8050.0	54.16
C	7800	7500	8400		7900.0	458.26
GG	250*					
P	2800*	2700*	2700*	3000*	2800.0	141.42
X	15800*	14100*	15700*		15200.0	953.94
M	7370	7110	7250		7243.3	130.13
Z	7730	7620	8150	8180	7920.0	286.71
AA	7500	7250	7300	7300	7337.5	110.87
BB	7850	13030*	8090		9656.7	2923.86
HH	7500	7500	6800	7300	7275.0	330.40

MEAN OF 98 MEASUREMENTS = 7843.3 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)

805.4 MICROGRAM/LITER = 10.3 PERCENT

STANDARD DEVIATION (MEAN)

81.4 MICROGRAM/LITER = 1.0 PERCENT

CONFIDENCE INTERVAL (95%)

163.5 MICROGRAM/LITER = 2.1 PERCENT

NOTE:

DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS. NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS USED BY A LABORATORY.

LAB	INTERLABORATORY TEST				SAMPLE A	TOC	
	MEASURED CONCENTRATION (MICROGRAM/LITER)				MEAN	ST.DEV.	
A1	44000	43500	45000	43500	44000.0	707.11	
A2	53000	53000	54000	52000	53000.0	816.50	
U1	47000	43500	43000	40500	43500.0	2677.06	
U2	46500	49500	49500	49000	48625.0	1436.14	
Y	52000	52000	52000	52000	52000.0		
EE	49000	47000			48000.0	1414.21	
FF	46800	45200	46800		46266.7	923.76	
GG	40300						

MEAN OF 26 MEASUREMENTS = 47676.9 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)
4054.9 MICROGRAM/LITER = 8.5 PERCENT

STANDARD DEVIATION (MEAN)
795.2 MICROGRAM/LITER = 1.7 PERCENT

CONFIDENCE INTERVAL (95%)
1638.2 MICROGRAM/LITER = 3.4 PERCENT

NOTE:

DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS.
NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS
USED BY A LABORATORY.

INTERLABORATORY TEST		SAMPLE B			TOC	
LAB	MEASURED CONCENTRATION (MICROGRAM/LITER)				MEAN	ST.DEV.
A1	41500	40000	41000	43000	41375.0	1250.00
A2	45000	46500	48500	47500	46875.0	1493.04
U1	41000	41500	40500	41000	41000.0	408.25
U2	43000	45500	45000	45000	44625.0	1108.68
Y	46000	46000	46000	45000	45750.0	500.00
EE	47000	44000			45500.0	2121.32
FF	46000	50000	48400	45200	47400.0	2203.03
GG	39800					

MEAN OF 27 MEASUREMENTS = 44403.7 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)
2840.6 MICROGRAM/LITER = 6.4 PERCENT

STANDARD DEVIATION (MEAN)
546.7 MICROGRAM/LITER = 1.2 PERCENT

CONFIDENCE INTERVAL (95%)
1123.9 MICROGRAM/LITER = 2.5 PERCENT

NOTE:
DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS.
NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS
USED BY A LABORATORY.

INTERLABORATORY TEST		SAMPLE C			TOC	
LAB	MEASURED CONCENTRATION (MICROGRAM/LITER)				MEAN	ST.DEV.
A1	49000	49500	52000	49500	50000.0	1354.01
A2	55000	56500	54000	55000	55125.0	1030.78
U1	53000	51500	49500	51000	51250.0	1443.38
U2	61000*	56500	56000	56000	57375.0	2428.13
Y	55000	55000	55000	55000	55000.0	
EE	51000	49000			50000.0	1414.21
FF	51600	50000	55200	51600	52100.0	2200.00
GG	50500					

MEAN OF 26 MEASUREMENTS = 52803.8 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)
2611.6 MICROGRAM/LITER = 4.9 PERCENT

STANDARD DEVIATION (MEAN)
512.2 MICROGRAM/LITER = 1.0 PERCENT

CONFIDENCE INTERVAL (95%)
1055.1 MICROGRAM/LITER = 2.0 PERCENT

NOTE:

DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS.
NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS
USED BY A LABORATORY.

INTERLABORATORY TEST		SAMPLE A			HYDCAR	
LAB	MEASURED CONCENTRATION (MICROGRAM/LITER)				MEAN	ST.DEV.
B	400	400	400	400	400.0	
C	800	1200	900		966.7	208.17
E	500	500	500	500	500.0	
F	1180					
G	3800	2100	4400	4400	3675.0	1087.43
H	2450	2280	2380	2520	2407.5	102.43
K	4100	3900	3900	4200	4025.0	150.00
L	3000	2400	2200	1800	2350.0	500.00
M	14200*	11300*			12750.0	2050.61
N	500	600			550.0	70.71
O	1750	1700			1725.0	35.36
Q1	500	500			500.0	
Q2	2900	2900			2900.0	
S	1800	1800	1800	1800	1800.0	
V	2000					
FF	7400*	3500			5450.0	2757.72
HH	700	700	500		633.3	115.47

MEAN OF 45 MEASUREMENTS = 1854.7 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)

1296.4 MICROGRAM/LITER = 69.9 PERCENT

STANDARD DEVIATION (MEAN)

193.3 MICROGRAM/LITER = 10.4 PERCENT

CONFIDENCE INTERVAL (95%)

389.4 MICROGRAM/LITER = 21.0 PERCENT

NOTE:

DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS. NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS USED BY A LABORATORY.

LAB	INTERLABORATORY TEST				SAMPLE 8		HYDCAR	
	MEASURED CONCENTRATION (MICROGRAM/LITER)				MEAN	ST.DEV.		
B	400	400	400	400	400.0			
C	400	400	400	400	400.0			
E	700	900	800	800	800.0	81.65		
F	410							
G	1100	1800	1600	2100	1650.0	420.32		
H	700	680	490	520	597.5	107.82		
K	1800	1800	1600	1300	1625.0	236.29		
L	1500	1100	1100	800	1125.0	287.23		
M	12200*	10900*			11550.0	919.24		
N	400	1000			700.0	424.26		
O	400	400			400.0			
Q1	250	250			250.0			
Q2	1300	1300			1300.0			
S	400	400	400	400	400.0			
V	2000							
FF	7000*	2000			4500.0	3535.53		
HH	500	400			450.0	70.71		

MEAN OF 45 MEASUREMENTS = 857.8 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)

561.6 MICROGRAM/LITER = 65.5 PERCENT

STANDARD DEVIATION (MEAN)

83.7 MICROGRAM/LITER = 9.8 PERCENT

CONFIDENCE INTERVAL (95%)

168.7 MICROGRAM/LITER = 19.7 PERCENT

NOTE:

DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS.
 NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS
 USED BY A LABORATORY.

LAB	INTERLABORATORY TEST				SAMPLE C		HYDCAR	
	MEASURED CONCENTRATION (MICROGRAM/LITER)				MEAN	ST.DEV.		
B	400	400	400	400	400.0			
C	400	400	400	400	400.0			
E	900	130	170	170	342.5	372.14		
F	420							
G	3500*	3200*	2800*	2900*	3100.0	316.23		
H	240	520	350	320	357.5	117.86		
K	1200	1200	1300	1400	1275.0	95.74		
L	1600	900	1200	700	1100.0	391.58		
M	11400*	4730*			8065.0	4716.40		
N								
O	400	400			400.0			
Q1	170	170			170.0			
Q2	1200	1200			1200.0			
S	400	400	400	400	400.0			
V	2000*							
FF	2400*	2000*			2200.0	282.84		
HH	100	100	100		100.0			

MEAN OF 38 MEASUREMENTS = 562.1 MICROGRAM/LITER

STANDARD DEVIATION (SINGLE MEASUREMENT)

423.8 MICROGRAM/LITER = 75.4 PERCENT

STANDARD DEVIATION (MEAN)

68.7 MICROGRAM/LITER = 12.2 PERCENT

CONFIDENCE INTERVAL (95%)

139.3 MICROGRAM/LITER = 24.8 PERCENT

NOTE:

DOUBLE LETTERS ARE CODES OF LABORATORIES OTHERWISE CODED BY SMALL LETTERS.
 NUMBERS COMBINED WITH LETTERS CHARACTERISE DIFFERENT ANALYTICAL METHODS
 USED BY A LABORATORY.

Mean and standard deviation of total of individual measurements

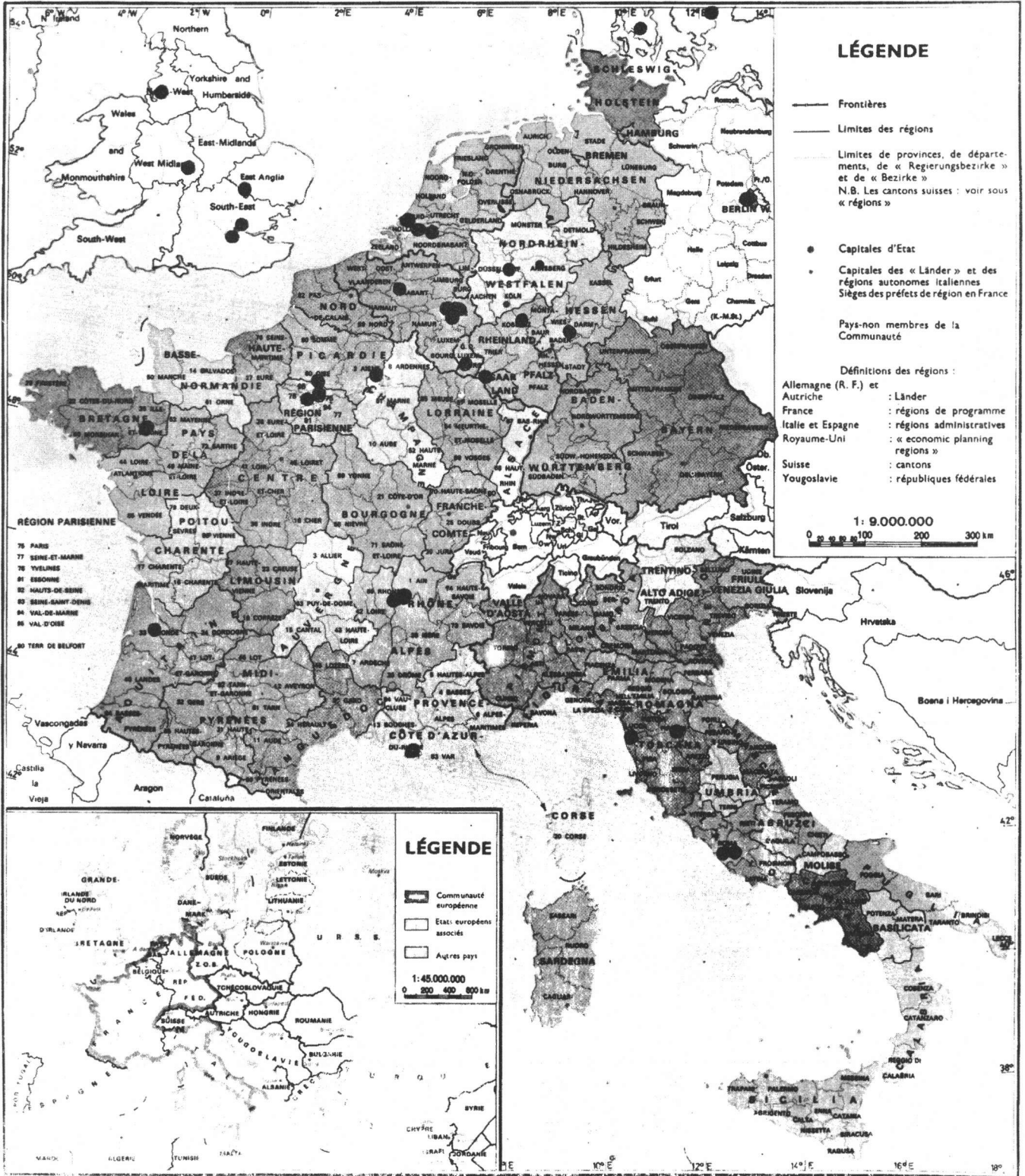
Parameter	Sample/ group	Number of accounted measurements	Number of outliers (NALIMOV test)	Mean \bar{x} (mg/l)	Standard deviation (mg/l)	Number of accounted measurements	Number of outliers ($\bar{x} \pm 4s$)	Mean \bar{x} (mg/l)	Standard deviation (mg/l)
Cyanides	A	80	22	0,095	0,054	82	20	0,100	0,062
	B	80	25	0,108	0,065	85 ⁺	20	0,106	0,071
	C	97	5	3,39	1,17	unchanged			
	A+B	160	47	0,101	0,060	167	40	0,103	0,066
	AI	29	2	0,052	0,033	unchanged			
	AII	51	20	0,119	0,048	53	18	0,126	0,058
	BI	30	2	0,054	0,041	unchanged			
	BII	50	23	0,140	0,055	55	18	0,134	0,068
	(A+B)I	59	4	0,053	0,037	unchanged			
	(A+B)II	101	43	0,129	0,052	108	36	0,130	0,063
	CI	9	-	2,50	0,98	unchanged			
CII	66	5	3,81	1,01	unchanged				
Phenols	A	92	17	7,80	0,88	97	12	7,87	1,17
	B	93	17	7,66	0,79	95	15	7,70	0,85
	C	98	13	7,84	0,80	99	12	7,82	0,83
	A+B	185	34	7,73	0,84	192	17	7,79	1,02
Hydro- carbons	A	45	3	1,86	1,30	unchanged			
	B	45	3	0,86	0,56	unchanged			
	C	38	9	0,56	0,42	45	2	0,89	0,90
TOC	A	26	-	47,7	4,1	unchanged			
	B	27	-	44,4	2,8	unchanged			
	C	26	1	52,8	2,6	27	-	53,1	3,0

⁺0,01 mg/l taken for non detectable values (NN)

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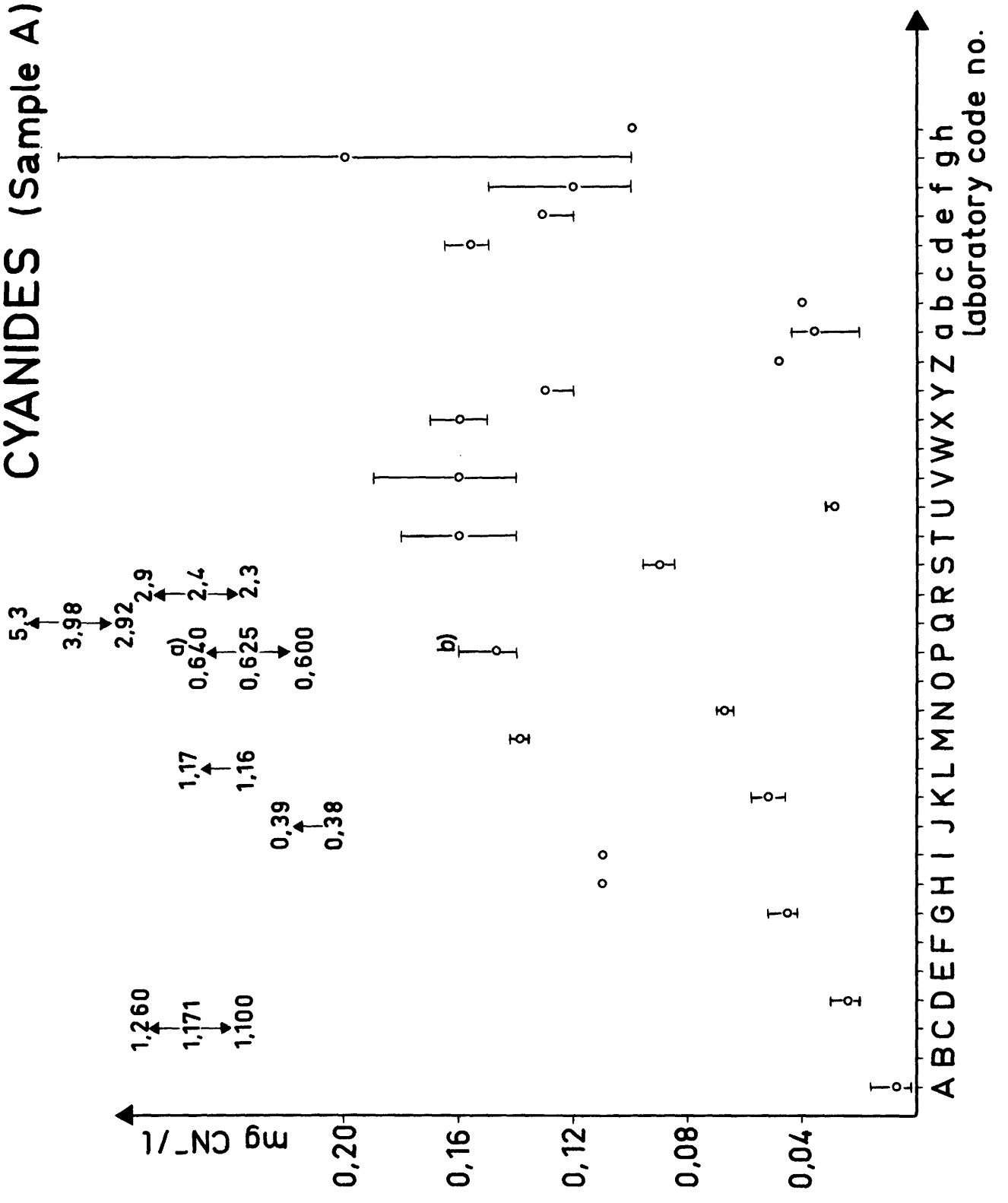
La Communauté européenne

RÉGIONS ET UNITÉS ADMINISTRATIVES



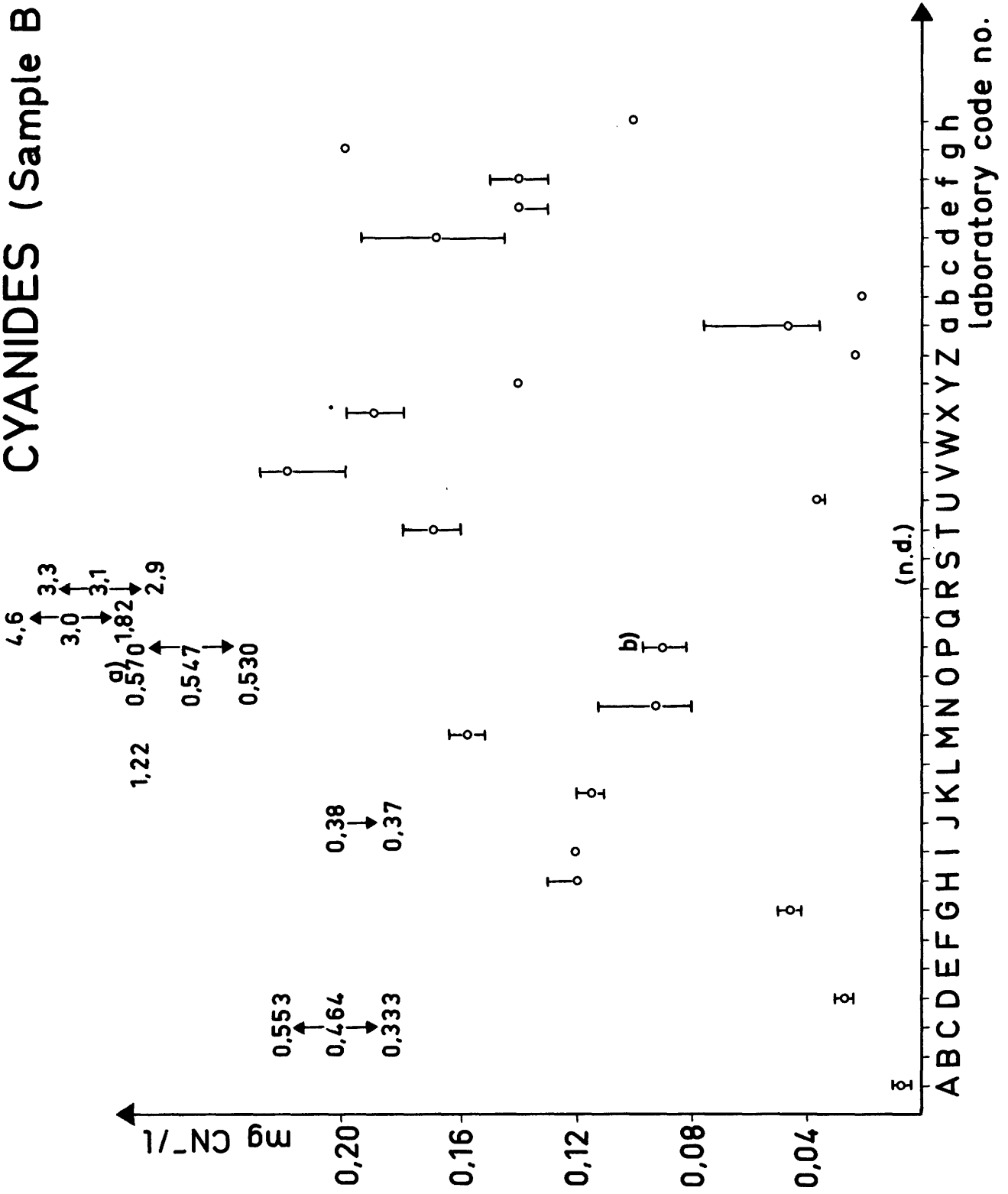
9.2.

CYANIDES (Sample A)



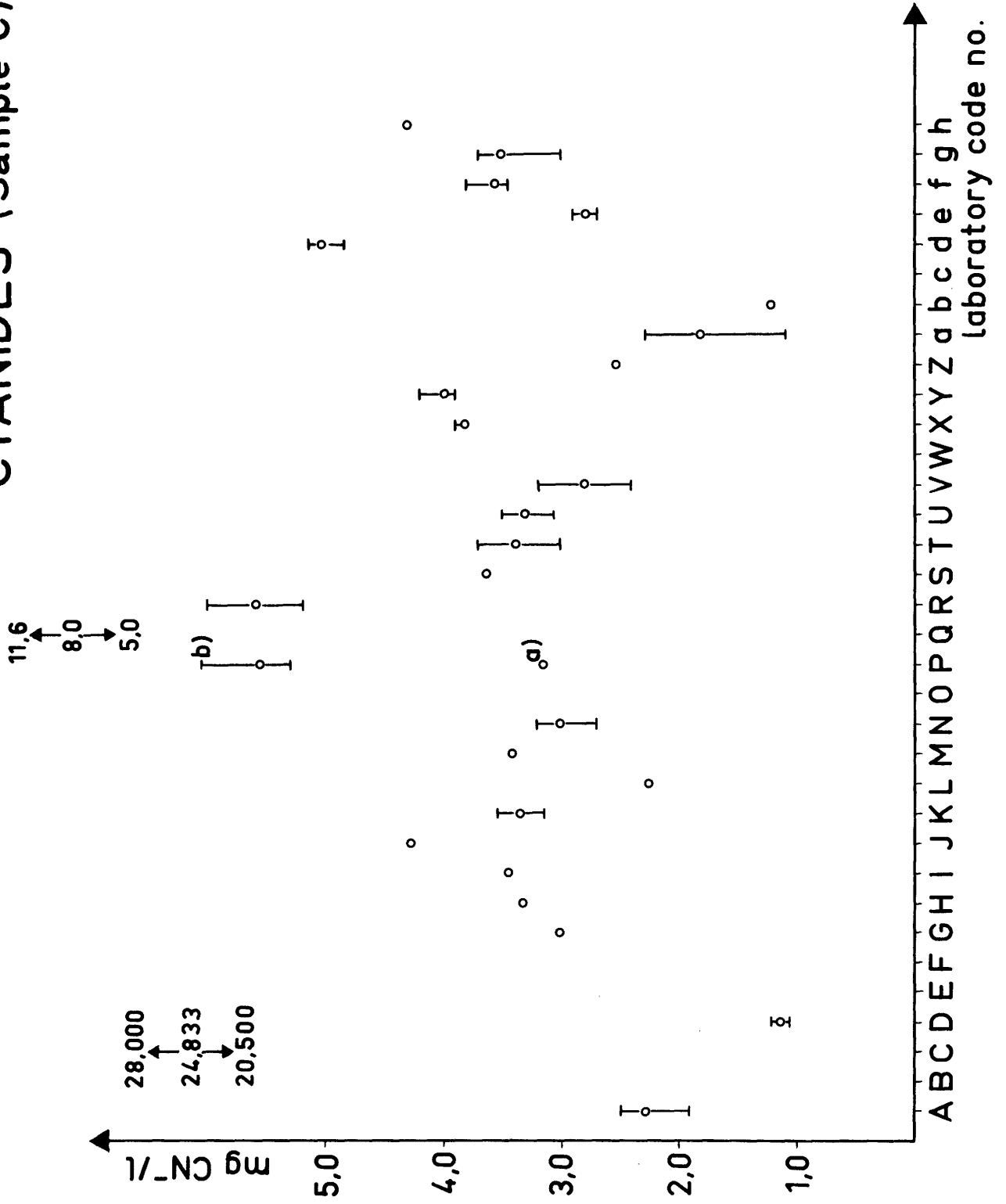
9.2.

CYANIDES (Sample B)

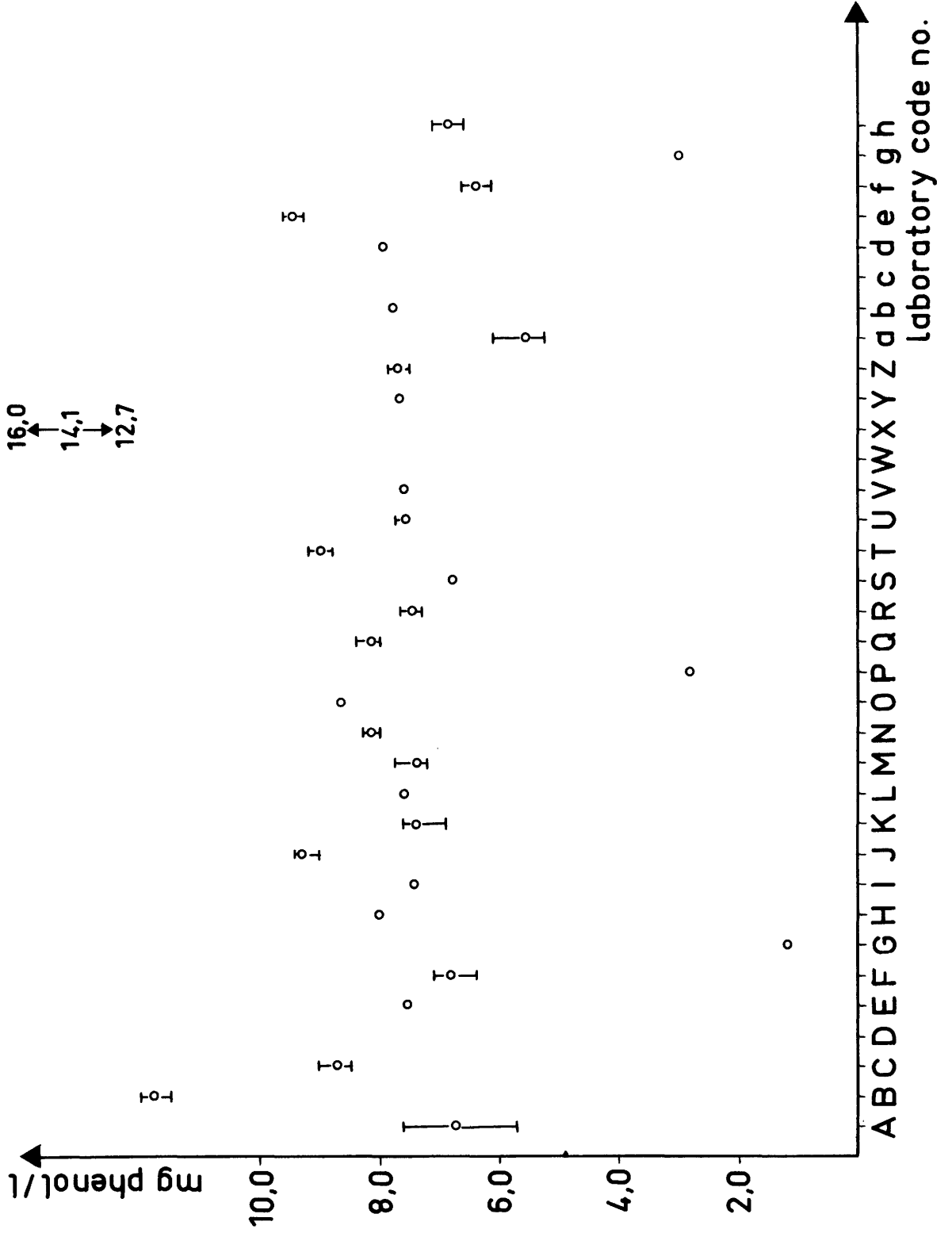


9.2.

CYANIDES (Sample C)

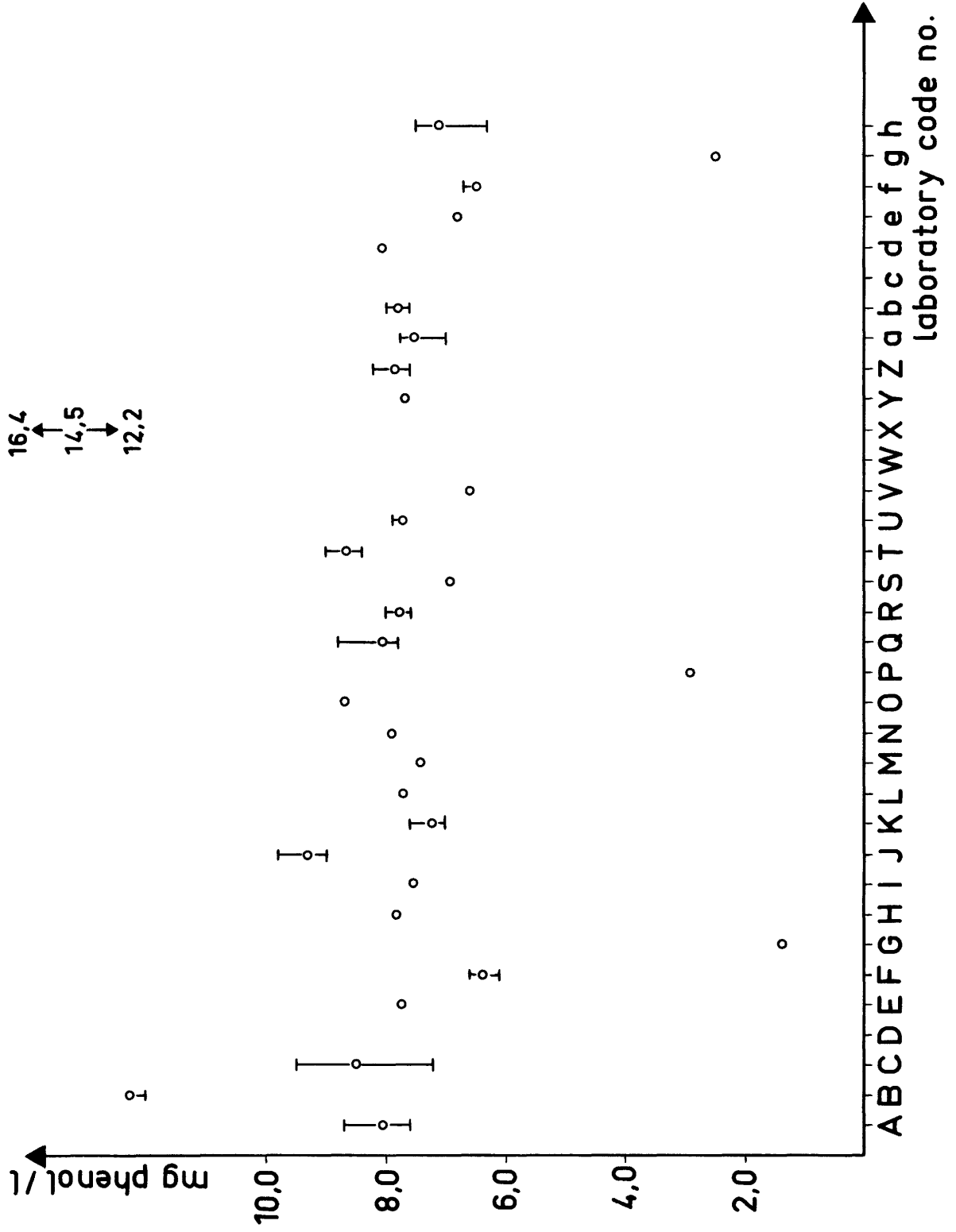


PHENOLS (Sample A)

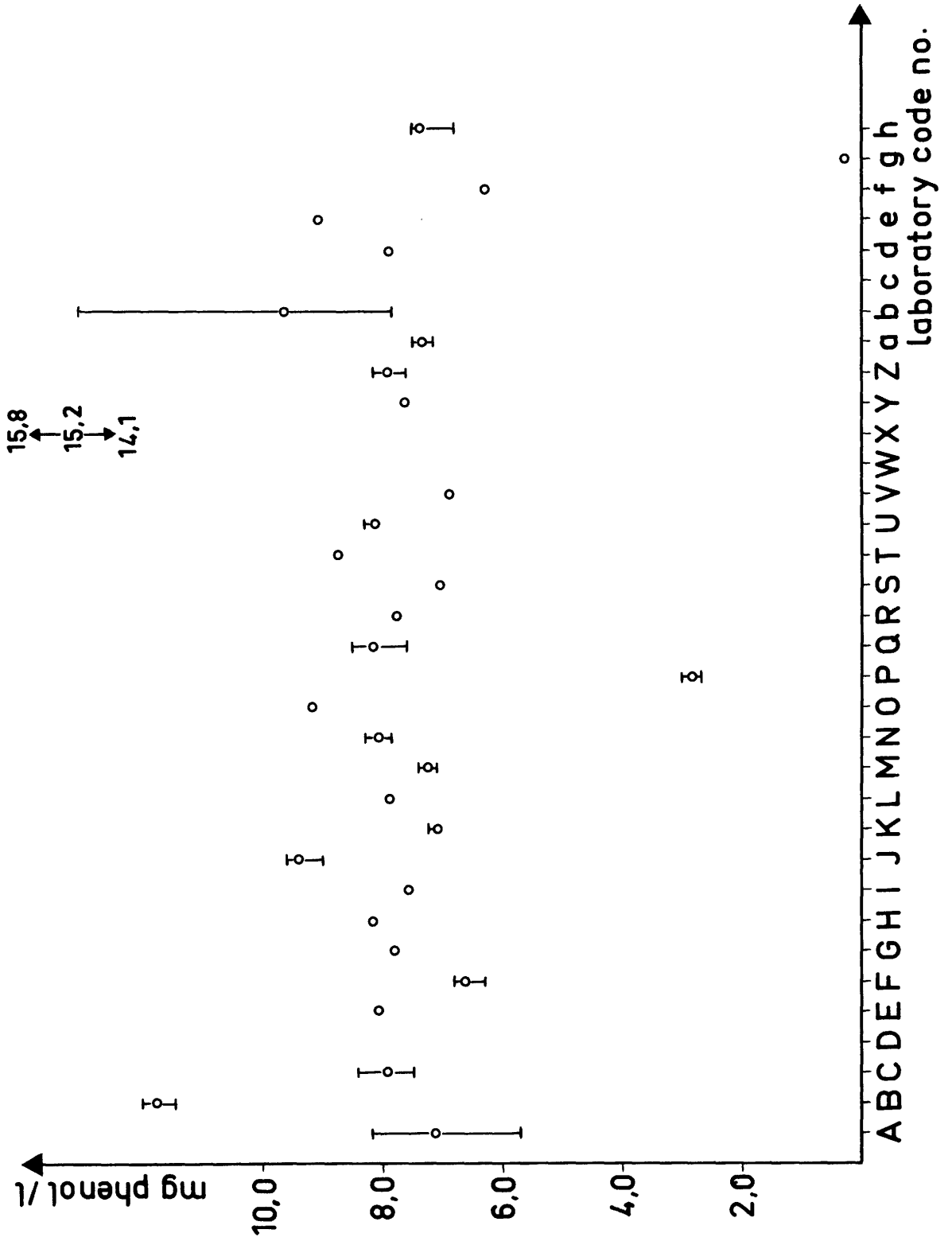


9.3.

PHENOLS (Sample B)

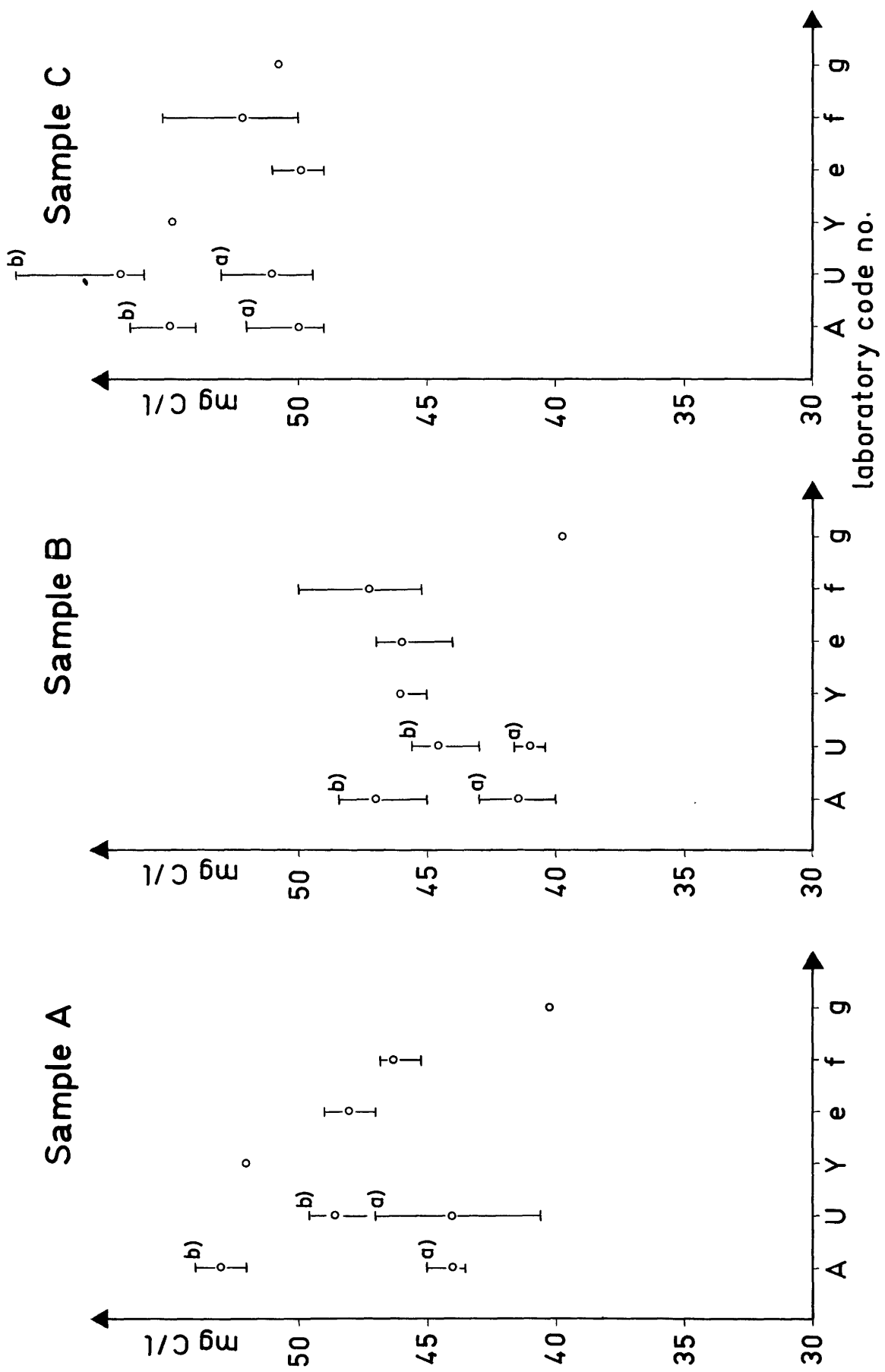


PHENOLS (Sample C)

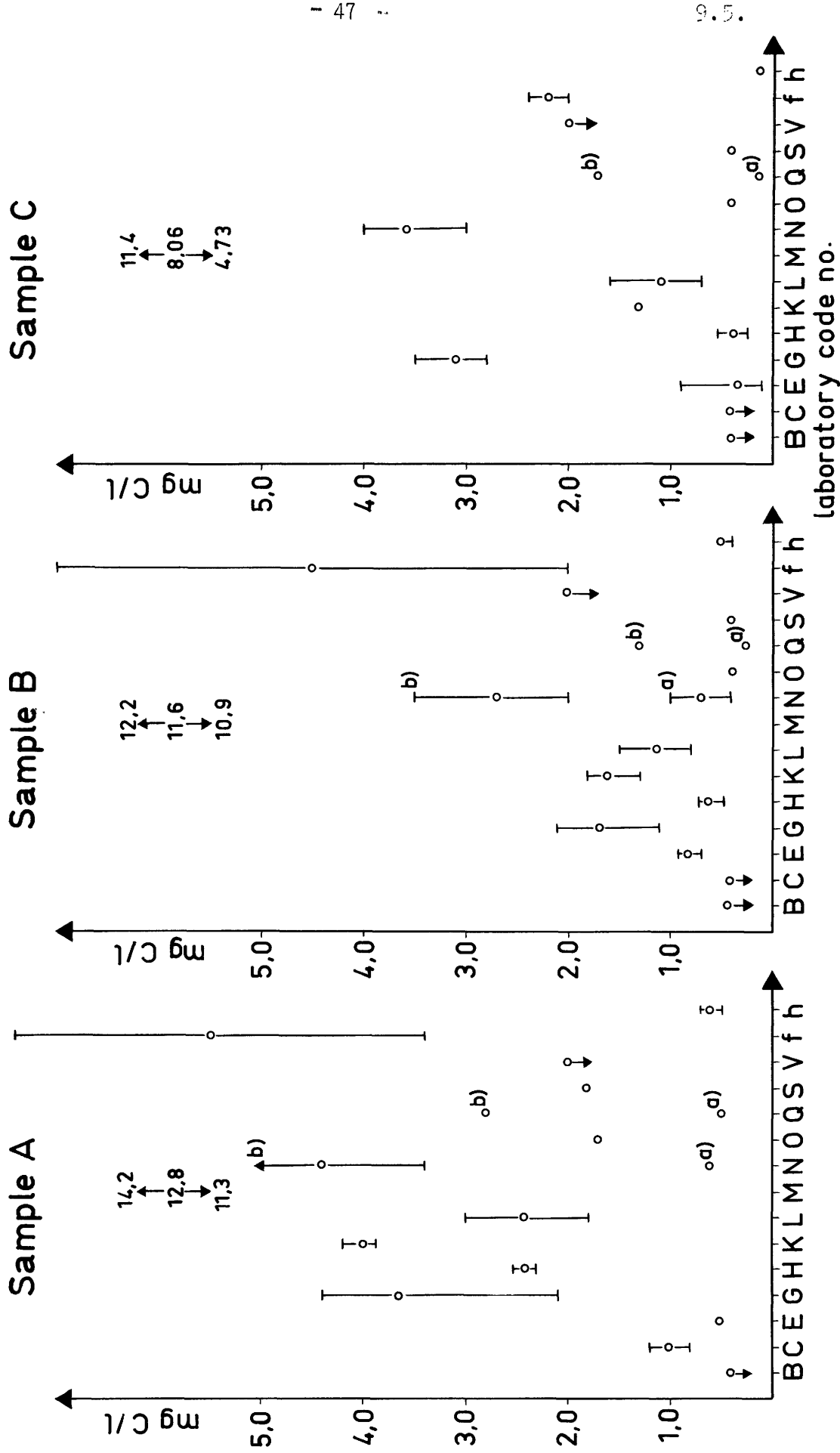


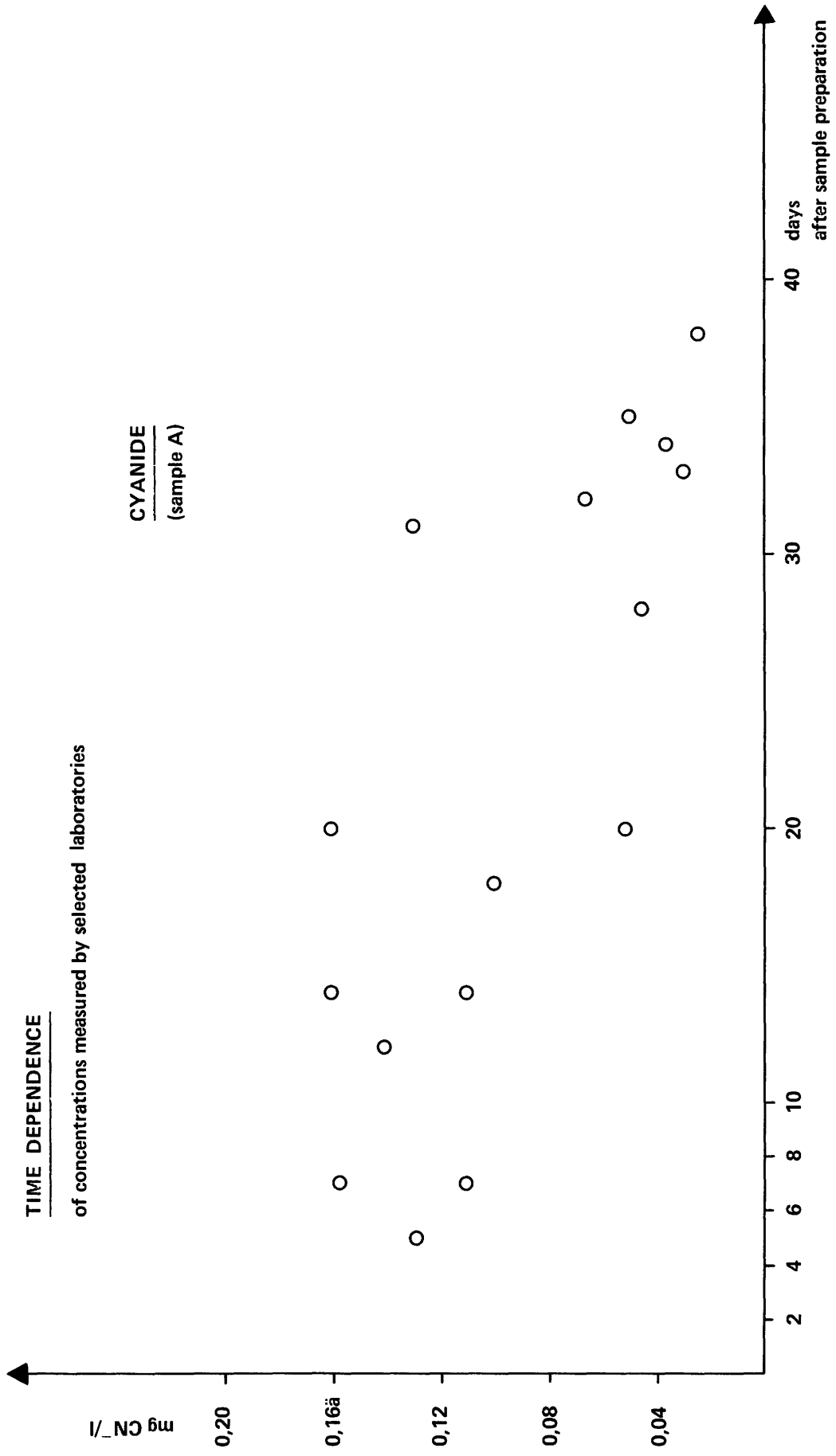
9.3.

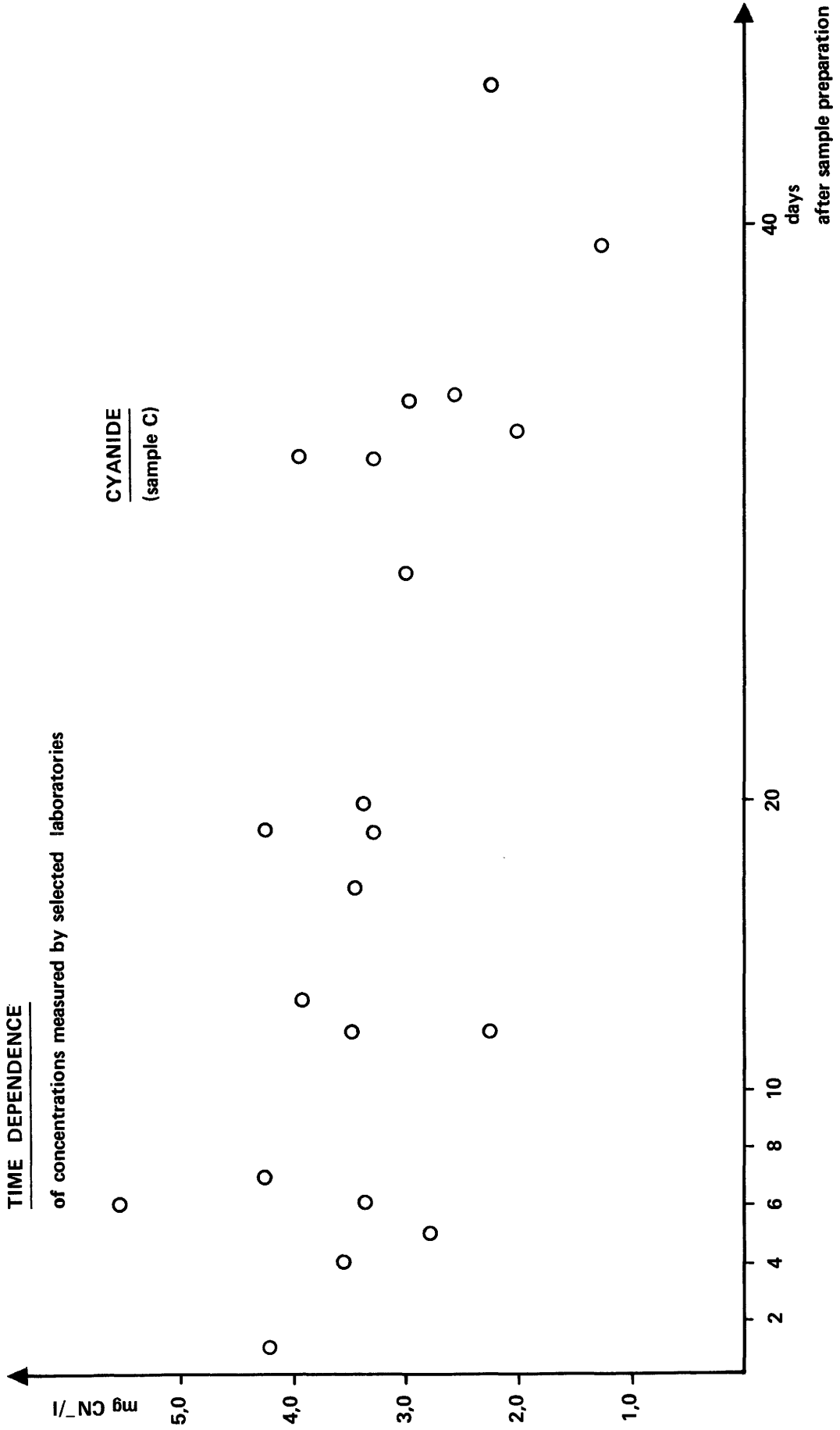
TOTAL ORGANIC CARBON

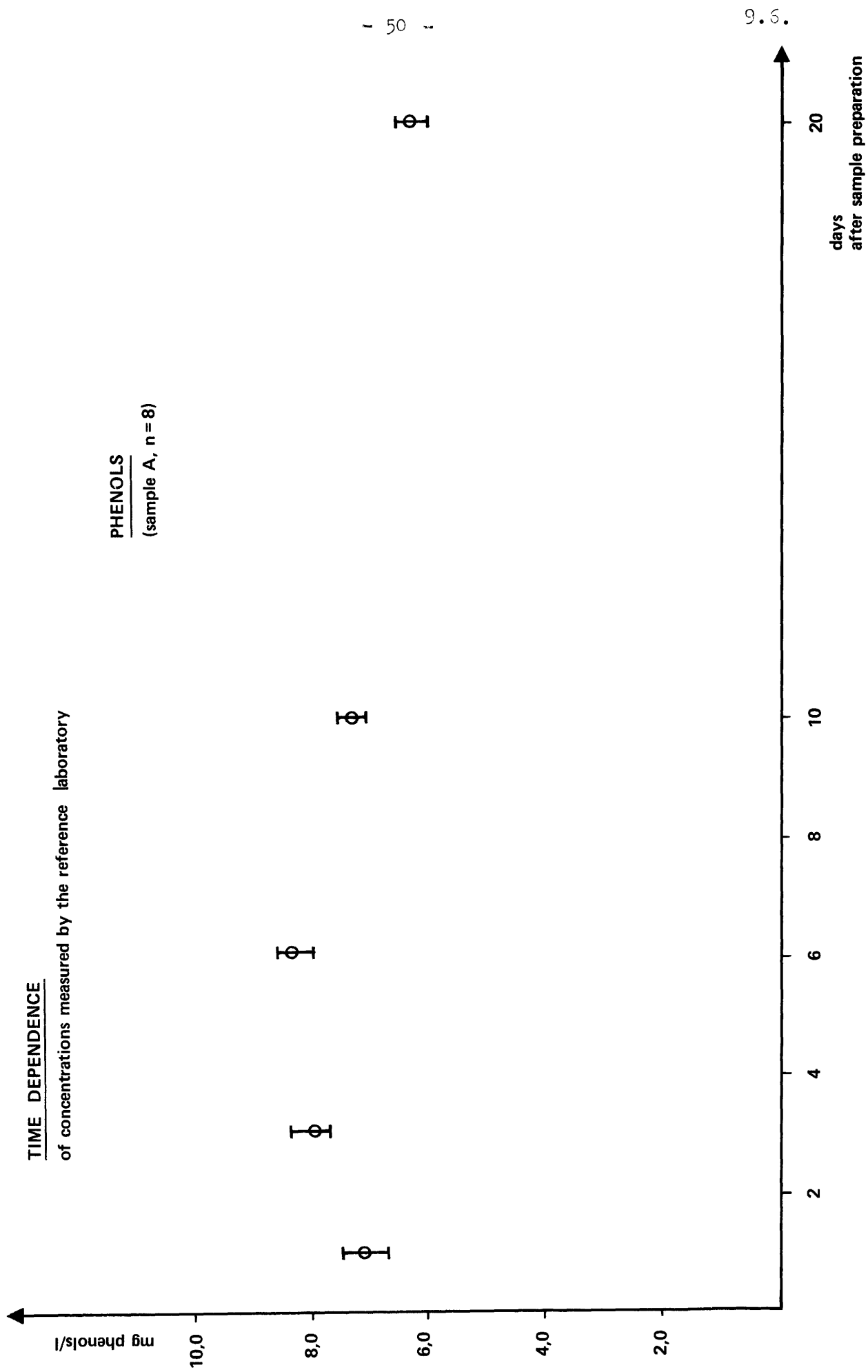


Aliphatic HYDROCARBONS





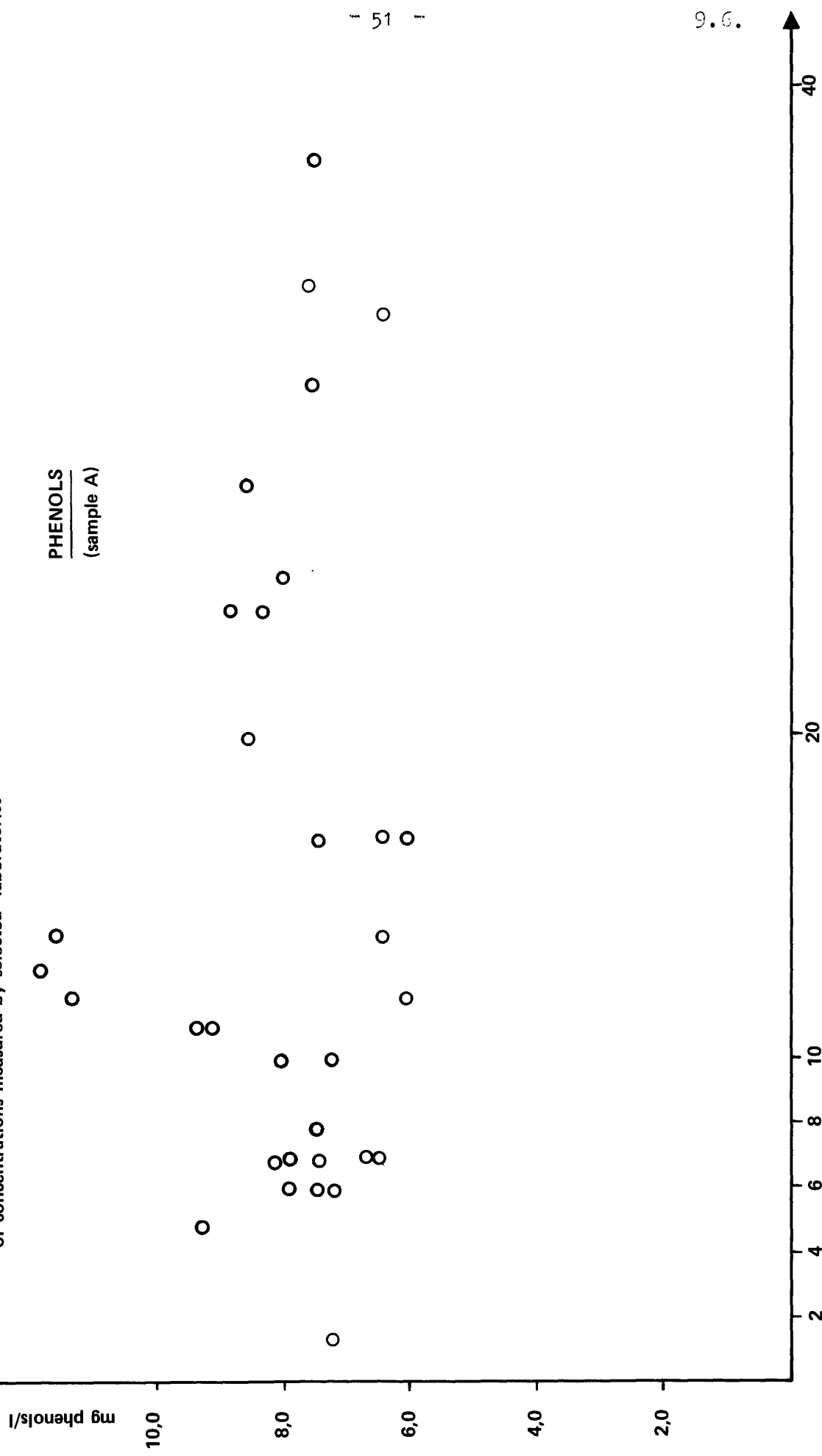




TIME DEPENDENCE

of concentrations measured by selected laboratories

PHENOLS
(sample A)



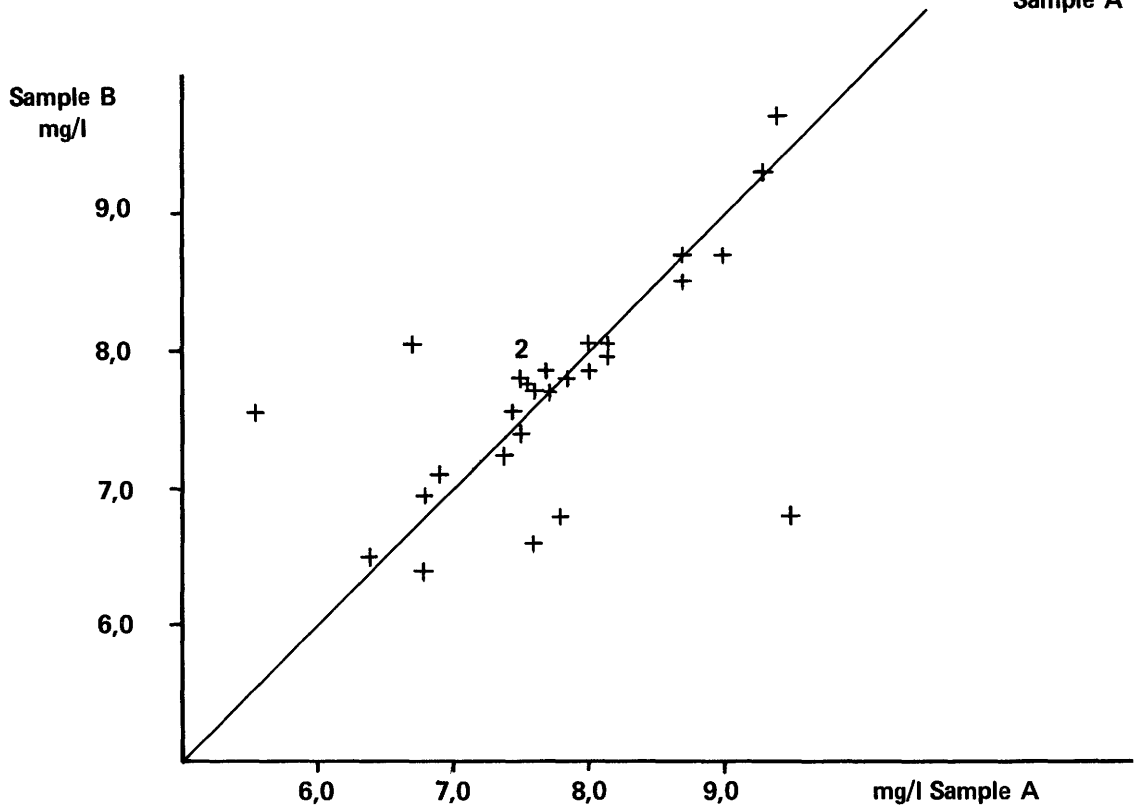
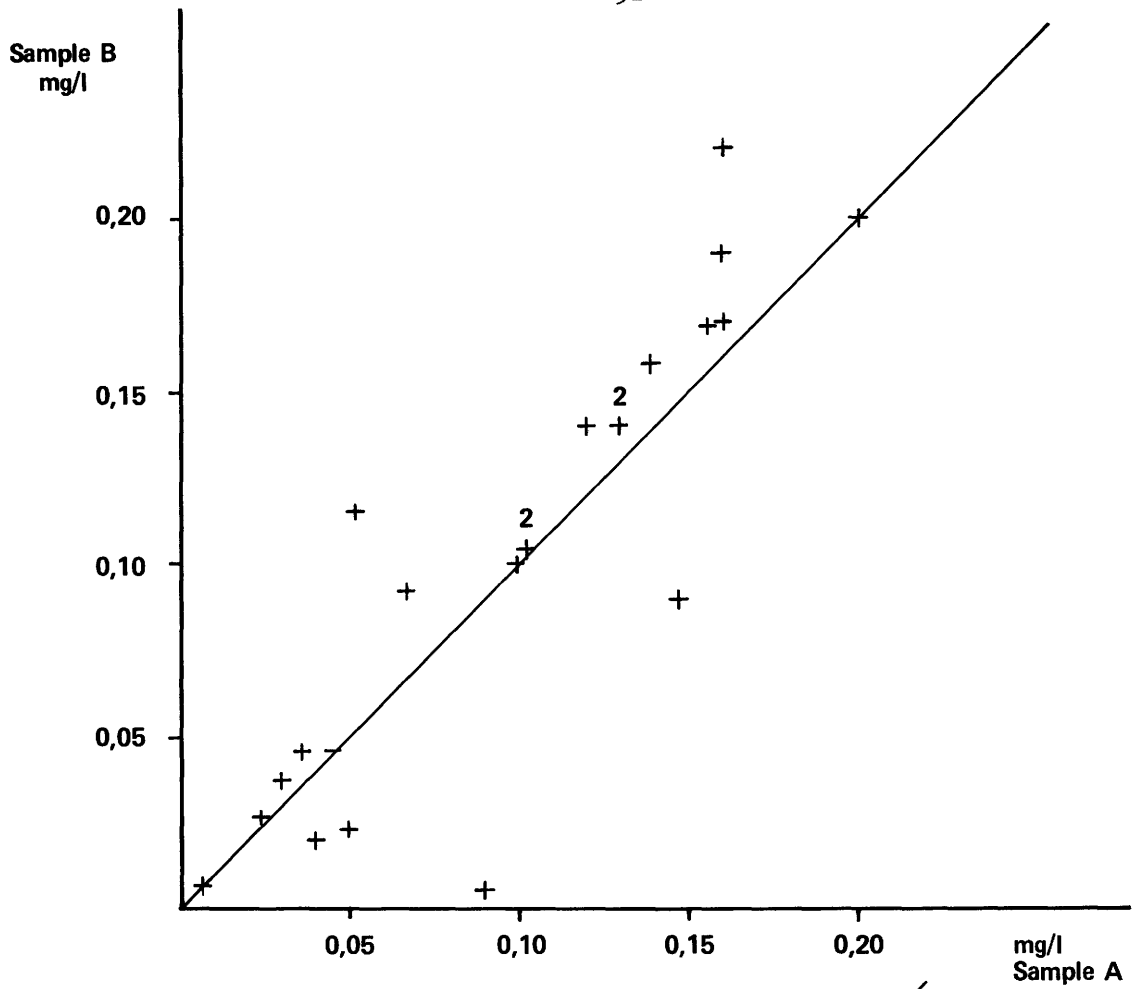


Fig. You den plot for samples A and B
a) Cyanides b) Phenols

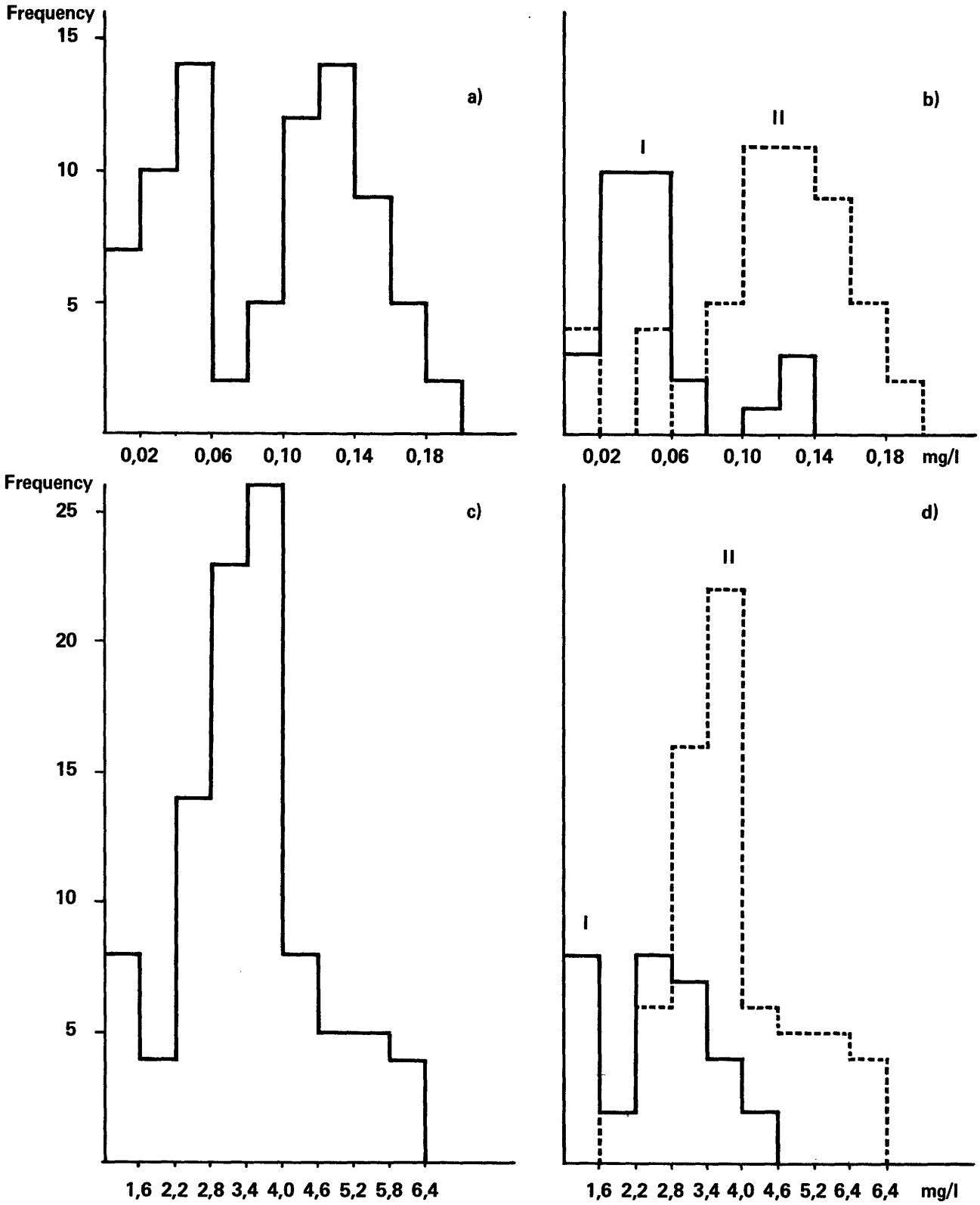


Fig. Frequency distribution of individual detominations of cyanides. a) Sample A. b) Sample A split into two subgroups I and II. c) Sample C. d) Sample C split into two subgroups I and II

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Directorate General
for Social Affairs

Luxembourg, 17 June 1974
VDV/jn

Health Protection
Directorate

Environment and Consumer
Protection Service

EXPLANATORY NOTE

on

A COLLABORATIVE EXERCISE ON THE DETERMINATION OF CYANIDE - PHENOLS
AND HYDROCARBONS IN SURFACE WATER SAMPLES

- I. In response to the wish expressed by the national experts at the meeting of 22 - 23 Jan. 1974, the Health Protection Directorate of the Directorate General for Social Affairs in collaboration with the Environment and Consumer Protection Service of the C.E.C. are organizing a collaborative exercise in regard to the quantitative measurement of cyanides, phenols and hydrocarbons in surface water samples. The aim of this programme is to investigate on Community level the comparability of measurement of results in order to facilitate the objective evaluation on a common base of the health hazards involved.

- II. The samples will be sent to the laboratory interested to participate at this exercise, not later than the beginning of September. The results have to be returned before the 5th of October. The discussion of results will take place in a meeting with the participating laboratories in Luxembourg on the 4th and 5th November 1974.

- III. Each sample will consist of 3 x 2 litres.

Sample "A" Synthetic "surface water" with unknown composition
Sample "B" Surface water with unknown composition
Sample "C" Surface water spiked with cyanide, phenols, hydrocarbons.

The three samples will be stabilised with NaOH (pH 11) to ensure conservation for cyanides and phenols.

You will be advised by telex of the date of flight sending the samples so as to be able to get them as soon as possible.

Because we know that transport over a long period of time could cause modifications in waters and dispersions of results, to obtain the most comparable results possible, please take care of additional parameters:

- 1) Control T° on reception: thermometer enclosed with the bottles in the box
- 2) Note the date of reception
- 3) Note exact date of analysis
- 4) Standard form for transmission of results will be enclosed in each box of samples.

IV. We kindly request the laboratories to closely adhere to the following points:

- 1) Each sample shall be investigated in quadruplicate (but minimum duplicate) i.e. starting from four separate samplings and analyzed by the same experienced worker for cyanides, phenols, hydrocarbons.
- 2) The results must be reported in mg/l without averaging.

The results should be sent before 5 October 1974 to:

Prof. Dr. K. AURAND
Institut für Wasser-, Boden- und Lufthygiene
des Bundesgesundheitsamtes
1000 Berlin 33
Postfach (B.R.D.)

using a standard form supplied.

S. JOHNSON

Environment and Consumer
Protection Service
C.E.C.
200, rue de la Loi
BRUXELLES (Belgique)

Dr. J. SMEETS

Division Environmental Hygiene
Health Protection Directorate
C.E.C.
Centre Louvigny
LUXEMBOURG (G.D.)

ANNEX I : Participants formular

ANNEX I

PARTICIPANTS FORMULAR

Our laboratory agrees to participate to the intercomparison programme "CYANIDE, PHENOLS and HYDROCARBONS in surface water samples"

- Name of Laboratory

- Address

- Telephone

- Person to contact

After compiling this, please send back to:

Dr. J. SMEETS

Division Environmental Hygiene
Health Protection Directorate
C.E.C.
Centre Louvigny
LUXEMBOURG (G.D.)

10.2. Note enclosed in the sample boxes

Prof. Dr.K. Aurand

Dr. M. Sonneborn

im
Institut für
Wasser-, Boden- und Lufthygiene
des Bundesgesundheitsamtes

Berlin-Dahlem, den 26.Aug.1974
Corrensplatz 1

To all participants of the Cyanides, Phenols and Hydrocarbons
Quality Control

Re: Collaborative Exercise on the Determination of Cyanide, Phenols
and Hydrocarbons in Surface Water Samples

The Commission of the European Communities, Luxembourg, has
charged us with the technical performance of above mentioned
study, and we hereby like to thank you once more for your
readiness of participating in this exercise. Having informed
you first by Explanatory Note of June 17th, 1974, we send you
today the samples to be studied.

Each sample will consist of 3 x 2 litres.

The three samples are stabilised with NaOH (pH 11) to ensure
conservation for cyanides and phenols.

Please take note of the following parameters:

- 1) Control maximum temperature during transport on reception.
You will find a maximum-thermometer in the box enclosed
with the bottles
- 2) Note date of reception
- 3) Note exact date of analysis
- 4) Please send the results of your analysis - without any
corrections - in the standard form, enclosed in the box,
up to October 5th, 1974.

The standard form of the results should be sent to

Prof. Dr. K. Aurand
Dr. M. Sonneborn
Institut für Wasser-, Boden- und Lufthygiene
des Bundesgesundheitsamtes
1 Berlin (W) 33
Corrensplatz 1

- 5) Each sample should be investigated in quadruplicate (but minimum duplicate) i.e. starting from four separate samplings and analysed by the same experienced worker for cyanides, phenols and hydrocarbons.
- 6) On receipt of the samples, moreover we kindly ask you to immediately send us in advance enclosed post card with the following datas:
 - I.) Date of arrival of the samples at your laboratory:
 - II.) Maximum temperature during transport on receipt:

Standard Form for Results of the collaborative study in EC-frame work on the determination of

Code N° of the laboratory	Cyanide, Phenols and Hydrocarbons												Calculated and expressed in reference to:
	Sample A			Sample B			Sample C						
	1)	2)	3)	4)	1)	2)	3)	4)	1)	2)	3)	4)	
Cyanide													mg/l CN ⁻
Phenols													mg/l C ₆ H ₅ OH (o-phenol)
Hydrocarbons													mg/l *
Date of Analysis													

Remarks: 1) 2) 3) 4): quadruplicate determination, minimum duplicate determination
Results with 2 or 1 decimal places according to your analytical sensitivity

*) C: organic carbon, without any corrections

Maximum of temperature during transport (as registered by the enclosed maximum-thermometer at arrival of the samples):

Date of reception of the samples:

Description of the Methods used and detection limit of the methods (exact description on separate sheets)

Questionnaire

EG collaborative study:

C y a n i d e s

1. Sample pretreatment

1.1. Treatment before distillation:

1.2. Distillation:

1.3. Treatment after distillation:

2. Analytical determination

2.1. Colorimetric determination:

Color reagent:

Incubation time:

Wave length of measurement:

Extraction of the color-phase:

2.2. Ionspecific electrodes:

2.3. Titrimetric determination:

Indicator:

2.4. Other methods:

3. Concentration range (the method is normally used for analysis of water samples):

4. Detection limit:

Code number:

Questionnaire

EG collaborative study:

P h e n o l s

1. Sample pretreatment

1.1. Extraction

1.2. Distillation:

1.3. Other methods:

2. Analytical determination

2.1. Colorimetric determination:

Color reagent:

Incubation time:

Wave length of measurement:

Extraction of the color-phase:

2.2. G-L-C:

2.3. Other methods:

3. Concentration range (the method is normally used for analysis of water samples):

4. Detection limit:

Code number:

Questionnaire

EG collaborative study:

H y d r o c a r b o n s

1. Sample pretreatment

1.1. Extraction:

1.2. Absorption by Florisil:

1.3. Other methods:

2. Analytical determination

2.1. Infrared-spectroscopy:

Calibration standard:

2.2. Other methods:

3. Concentration range (the method is normally used for analysis of water samples):

4. Detection limit:

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11. Selected Literature (cit. by the participating Laboratories)

11.1. Cyanides

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11.2. Phenols

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Tour Europe - AFNOR, Paris (1973)

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Ges. Deutscher Chemiker, Weinheim (1973)

Standard Methods for the Examination of Water and Waste-
water, American Public Health Association, New York (1971)

11.3. Hydrocarbons

Association Française de Normalisation

Tour Europe - AFNOR, Paris (1973)

Deutsche Einheitsverfahren zur Wasseruntersuchung,

Ges. Deutscher Chemiker, Weinheim (1973)

Specific descriptions of the methods used by the participating laboratories are compiled at the Health Protection Directorate, European Communities, Luxembourg.