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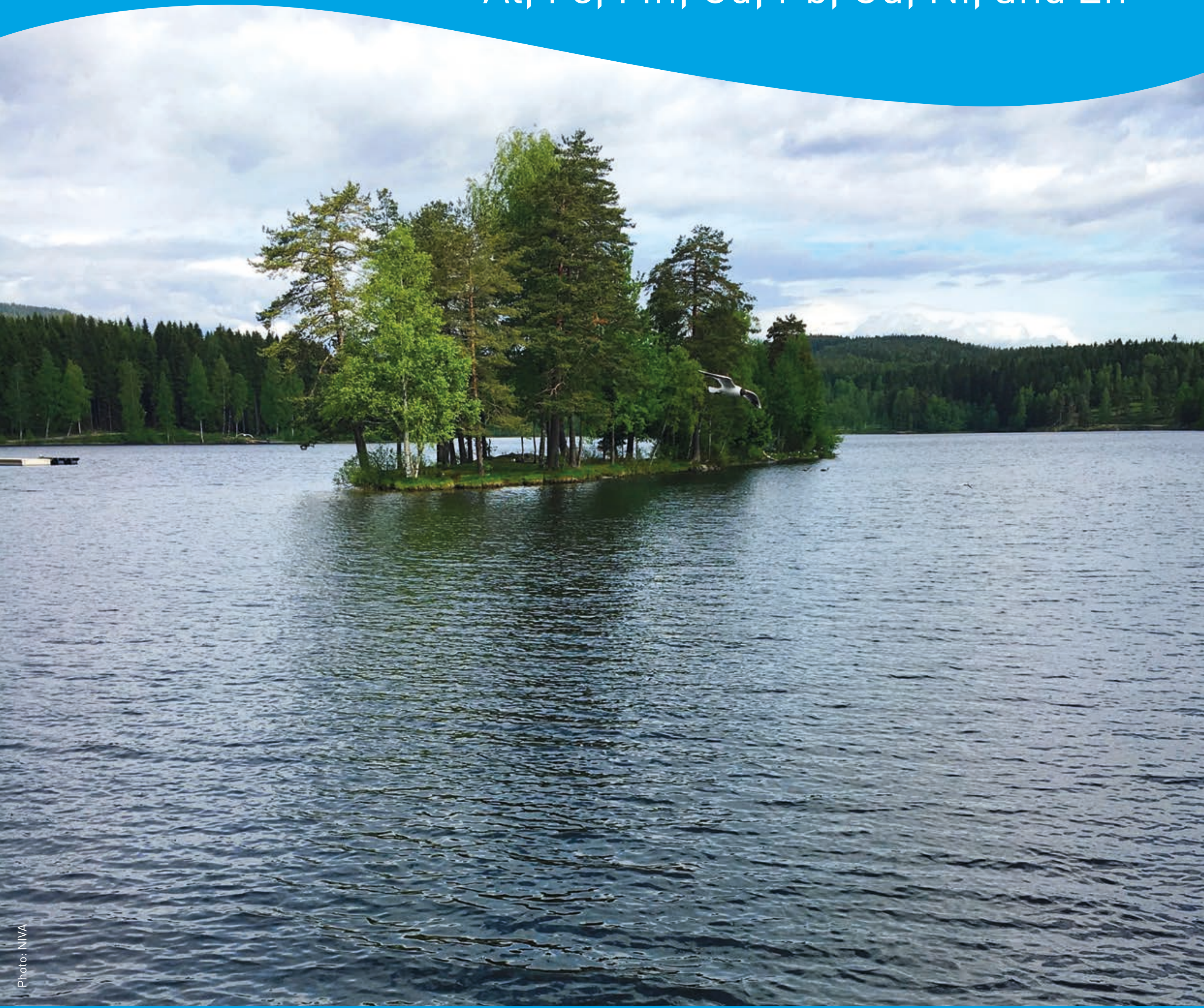
ICP Waters Report 134/2017

Intercomparison 1731: pH, Conductivity, Alkalinity, NO3-N, Cl, SO4, Ca,Mg, Na, K, TOC, Al, Fe, Mn, Cd, Pb, Cu, Ni, and Zn



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International Cooperative Programme on Assessment and Monitoring Effects of Air Pollution on Rivers and Lakes

Convention on Long-Range Transboundary Air Pollution

LRTAP Long-range Transboundary Air Pollution

Norwegian Institute for Water Research

REPORT

Main Office
Gaustadalléen 21
NO-0349 Oslo, Norway
Phone (47) 22 18 51 00
Telefax (47) 22 18 52 00
Internet: www.niva.no

NIVA Region South
Jon Lilletuns vei 3
NO-4879 Grimstad, Norway
Phone (47) 22 18 51 00
Telefax (47) 37 04 45 13

NIVA Region East
Sandvikaveien 59
NO-2312 Ottestad, Norway
Phone (47) 22 18 51 00
Telefax (47) 62 57 66 53

NIVA Region West
Thormøhlensgate 53 D
NO-5006 Bergen Norway
Phone (47) 22 18 51 00
Telefax (47) 55 31 22 14

NIVA Denmark
Ørestads Boulevard 73
DK-2300 Copenhagen
Phone (45) 8896 9670

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Summary

88 laboratories were invited to participate in the current intercomparison. Of these, 38 from 21 different countries accepted the invitation and all of them submitted results to the Organization. Two sample sets were prepared: one for the determination of major ions and one for heavy metals. Based on the general target accuracy of ± 20% or the special accuracy limit for pH and conductivity (± 0.2 pH units and ± 10% respectively), 76% of the overall results were considered acceptable. This is in line with previous editions. The best results were reported for the analytical variables: sulphate, magnesium, manganese, cadmium, copper, nickel and zinc with acceptance rate of 90% or higher. For pH, 53 % of the reported results fulfilled the acceptance criteria. Harmonization of the analytical methods used and of the practical procedures followed, may be the most important way to improve the comparability for these parameters.

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1. Sammenligning	1. Intercomparison
2. Sur nedbør	2. Acid precipitation
3. Kvalitetskontroll	3. Quality Control
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Carlos Escudero-Oñate	Øyvind Garmo	Heleen de Wit
Project Manager	Quality assurance	Research Manager

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CONVENTION ON LONG-RANGE TRANSBOUNDARY AIR POLLUTION

INTERNATIONAL COOPERATIVE PROGRAMME ON ASSESSMENT AND MONITORING EFFECTS OF AIR POLLUTION ON RIVERS AND LAKES

Intercomparison 1731: pH, Conductivity, Alkalinity, NO3-N, Cl, SO4, Ca, Mg, Na, K, TOC, Total-P, Al, Fe, Mn, Cd, Pb, Cu, Ni and Zn

Prepared at the ICP Waters Programme Centre
Norwegian Institute for Water Research
Oslo, November 2017

Preface

The International Cooperative Programme on Assessment and Monitoring Effects of Air Pollution on Rivers and Lakes (ICP Waters) was established under the Executive Body of the UNECE Convention on Long-range Transboundary Air Pollution (LRTAP) in July 1985. Since then, ICP Waters has been an important contributor to document the effects of implementing the Protocols under the Convention. Numerous assessments, workshops, reports and publications covering the effects of long-range transported air pollution have been published over the years.

The ICP Waters Programme Centre is hosted by the Norwegian Institute for Water Research (NIVA), while the Norwegian Environment Agency leads the programme. The Programme Centre's work is supported financially by the Norwegian Environment Agency.

The objective of the Programme is to establish an international network of surface water monitoring sites and promote international harmonization of monitoring practices. One of the aims is to detect long-term trends in effects of acidic deposition on surface water chemistry and aquatic biota, and to reveal the dose/response relationship between water chemistry and aquatic biota.

One of the tools in this work is inter-laboratory quality assurance tests. The bias between analyses carried out by the individual participants of the Programme has to be clearly identified and controlled.

We hereby report the results from the 31st intercomparison of chemical analysis.

Carlos Escudero-Oñate

ICP Waters Programme Centre
Oslo, November 2017

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Summary

The Intercomparison was organized as part of the between-laboratory quality control programme, as stated in "Manual for Chemical and Biological Monitoring" (1), by the International Cooperative Programme on Assessment and Monitoring of Acidification in Rivers and Lakes (ICP Waters).

The intercomparison was performed in the period April - September 2017, and included the determination of major ions and metals in natural water samples. The participants were invited to determine pH, conductivity, alkalinity, nitrate, chloride, sulphate, calcium, magnesium, sodium, potassium, total organic carbon, total phosphorous, aluminium, iron, manganese, cadmium, lead, copper, nickel and zinc.

Two sample sets were prepared for this intercomparison, one for the determination of the major ions (plus TOC and Total-P) and one for the heavy metals. 88 laboratories were invited to participate, and samples were sent to the 38 laboratories who accepted. All of them submitted results to the Programme Centre before the final statistical treatment of the data. 21 countries are represented in the current intercomparison program.

The median value of the results received from the participants for each variable was selected as "true" value. On average 76% of the result pairs were considered acceptable, the target limit being the median value ± 20%, except for pH and conductivity, where special acceptance limits were selected, ± 0.2 pH units and ± 10 %, respectively.

For pH, the accuracy limit was, as in earlier intercomparisons, extended from the target acceptance limit of ± 0.1 units to ± 0.2 units, and 53 % of the result pairs were acceptable when using this extended limit. A total error of ± 0.2 units for pH measurements, therefore seems to be a more reasonable basis for the assessment of the accuracy between laboratories than the target limit of ± 0,1 units.

The best results in terms of acceptance were obtained for sulphate, magnesium, manganese, cadmium, copper, nickel and zinc with 90% or more of the results accepted.

Good quality was observed as well for the reported conductivity data; 77% of the results provided by the participants fulfilled the target accuracy.

A novelty in the current edition is the addition of Total-P as a new variable in the sample set AB. From the 38 laboratories that accepted the invitation to join the intercomparison, half (19) provided results for Total-P. From these, just 4 results (21%) where considered acceptable according to the target accuracy set for this variable.

1 Introduction

The international cooperative programme on assessment and monitoring of effects of air pollution on rivers and lakes (ICP Waters) was established under the Executive Body of the UNECE Convention on Long-range Transboundary Air Pollution (LRTAP) in July 1985. Since then ICP Waters has been an important contributor to document the effects of implementing the Protocols under the Convention. Numerous assessments, workshops, reports and publications covering the effects of long-range transported air pollution has been published over the years.

ICP Waters operates from the middle of a monitoring hierarchy that is designed to evaluate the environmental effects of air pollutants on surface waters chemistry and biology, and predict future ecosystem changes occurring under different deposition scenarios. Lower in the hierarchy is a series of national networks that employ progressively less comprehensive and frequent sampling but greater spatial coverage, culminating in one-time regional surveys. Achieving the Programme objectives requires that both the temporally intensive and regionally extensive data are collected on a continually basis.

As stated in the "ICP Waters Programme Manual" (1), between-laboratory quality control is necessary in a multilaboratory programme to assure clear identification and control of the bias between analyses carried out by individual participants of the Programme. Such biases may arise by use of different analytical methods, errors in the laboratory calibration solutions or through inadequate within-laboratory control.

The between-laboratory control carried out by the Programme Centre is based on the "round robin" concept and the procedure of Youden (2, 3), which is briefly described in Appendix C. This thirty-first intercomparison test, called 1731, included the determination of the major components and metal ions in natural water samples: pH, conductivity, alkalinity, nitrate, chloride, sulphate, calcium, magnesium, sodium, potassium, total organic carbon, total phosphorous aluminium, iron, manganese, cadmium, lead, copper, nickel and zinc.

2 Accomplishment of the intercomparison

The preparation of the sample solutions that were delivered to the different participating laboratories is presented in Appendix B of this document. At the Task Force meeting in Burlington, Canada, in October 2009, it was decided that, as earlier, two sample sets should be included in this intercomparison, one sample pair for the determination of the major ions and one for heavy metals. It was decided that total organic carbon and aluminium should also be included. Recently it was also decided to include Total-P as additional variable.

The samples were shipped from the Programme Centre the week 25 of 2017. With some exceptions, the participants received the samples within one week. Despite samples were sent with a declaration of absence of commercial value and description of only testing samples, in some cases, delays in the reception of the samples were reported by the laboratories. Further research in the origin of the trouble demonstrated that delay was due to troubles in the customs in some of the countries.

To ensure the integrity and minimal degradation of the samples, participants were encouraged to analyze them as soon as possible and save their analytical results in the Organization's database as soon as possible.

3 Discussion

The general rule for target accuracies, outlined in the Manual for Chemical and Biological Monitoring (1), shall normally be used as acceptance limits for the results of the intercomparison test. These limits correspond to either the detection limit of the method, or 20 % of the true value, whichever being the greater, i.e. fixed or relative acceptance limits.

In Table 1 an evaluation of the results of intercomparison 1731 is presented with the number and percentage of acceptable results based on the target accuracy (except for pH and conductivity). In Appendix D, Table 4, the individual results of each laboratory are presented. Some laboratories use far more digits than are statistically significant. This is unnecessary, and each laboratory should determine how many digits are significant for each of their analytical methods. It is however acceptable to report results with one digit more than is statistically significant as this will reduce the round-off error in the statistical calculations.

In this edition 38 laboratories submitted results to the intercomparison. If results for the different variables are averaged, 76 % of them were located within the general target accuracy of $\pm$ 20 %, or the special accuracy limit for pH and conductivity ($\pm$ 0.2 pH units and $\pm$ 10% respectively). This result is in line with previous editions. As previously stated, the best acceptance (≥90%) was observed in the determination of sulphate, magnesium, manganese, cadmium, copper, nickel and zinc.

The lowest acceptable results were reported for alkalinity (17%) and Nitrate+nitrite-N (35%). In the case of pH, the relatively low percentage of acceptable results can be partially explained in basis of the transformation that undergoes the actual variable (molar concentration of H^+). Such a transformation provokes that slight differences in H^+ concentration in solutions in regions close to neutrality lead to a relatively large numerical difference when applying the -log operator. In addition to the aforementioned, pH results may be strongly affected by the method used. This problem has been demonstrated through several earlier intercomparisons, and will remain a problem as long as different methods, different working procedures and different instrumental equipment for pH determination are used by the participating laboratories. The samples will also be exposed to different temperature and travel time during shipment. A total error of $\pm$ 0.2 pH units seems to be a reasonable assessment of the accuracy for pH measurements, when near neutral water samples - which are not at CO_2 equilibrium - are analyzed.

Due to the high precision of the reported results for conductivity in earlier intercomparisons, from the 2012 edition the Organization decided to reduce the acceptance limit for this analytical variable from the target value of ± 20 % to $\pm$ 10 % and this criterion was still used in the current one.

It has to be taken into account that despite samples have been spiked and then, the concentrations of some of the variables are still higher than could be expected in natural samples, some of the laboratories do not have available methods sensitive enough to determine heavy metals at trace level.

As it had been observed in the last years, the current edition confirms that plasma techniques (ICP-AES and ICP-MS) are taking over for atomic absorption methods, which were the dominating methods some years ago. There's also a general trend to use ICP-MS instead of ICP-AES for the determination of trace heavy metals.

The low fraction of acceptable results in the determination of some of the variables may in some cases be explained by either rather low concentration, compared to the methods that have been used, or that the samples were not sufficiently stable. When the concentrations are close to the detection limits of the methods used by the participants, it is expected that the spread of the results will be greater than ± 20 %. The laboratories which reported results outside this limit should improve their methods to obtain a better accuracy and then be able to get a better score in the intercomparison assay. In general terms the use of some analytical methods seems to be less suited for the water samples analyzed in this programme, as the detection limits of some methods applied by participants are too high. This is especially true for some manual methods, and some of the methods used for the determination of metals, especially when the concentration is very low. It is important that methods with detection limits low enough are used by the participating laboratories.

It should be further discussed which concentration levels for the heavy metals would be most useful for ICP Waters in the coming intercomparisons as well as whether *absolute* acceptance limits should be used instead of the *relative* one (± 20 %), which is used in this intercomparison, in cases where the results are close to the detection limit. In such cases, it is important that the steering committee decides the target detection limit that should be achieved by the participating laboratories.

Table 1. Evaluation of the results from intercomparison 1731.

				Acceptable Limit	Number of pairs		Acceptable results for intecalibration (%)			
Variable	**Sample pair**	**Sample 1**	**Sample 2**	**%**	**Total**	**Accept.**	**1731**	**1630**	**1529**	**1428**
pH	AB	5.78	5.76	3.6	32	17	53	56	64	68
Conductivity,	AB	2.9	2.55	10	31	24	77	77	89	93
Alkalinity,	AB	0.022	0.020	20	23	4	17	46	75	26
NO3+NO2-N	AB	66	61	20	26	9	35	71	88	14
Chloride,	AB	2	1.77	20	28	23	82	87	97	93
Sulphate,	AB	5.62	5	20	29	26	90	90	97	87
Calcium,	AB	2.40	2.09	20	30	25	83	93	97	97
Magnesium,	AB	0.37	0.32	20	30	28	93	89	100	87
Sodium,	AB	1.63	1.44	20	29	25	86	96	97	97
Potassium,	AB	0.23	0.21	20	29	20	69	86	97	97
TOC	AB	15.7	14.3	20	21	17	81	81	70	82
Total P	AB	11.5	10	20	19	4	21	-	-	-
Aluminium,	CD	178	157	20	22	18	82	75	89	78
Iron,	CD	81.8	73.1	20	23	17	74	87	81	74
Manganese,	CD	48.9	43	20	23	23	100	84	84	88
Cadmium,	CD	9.68	8.59	20	24	22	92	90	100	84
Lead,	CD	7.82	6.82	20	24	21	88	86	77	80
Copper,	CD	29.6	27.0	20	22	21	95	86	93	88
Nickel,	CD	14.3	12.7	20	23	23	100	90	97	92
Zinc,	CD	19.7	18.4	20	23	22	96	77	83	79
Total					511	389	76	(81)	(88)	(80)

Units: Conductivity: mS/m
Alkalinity: mmol/l
Nitrate+nitrite-N: µg N/l
Total P: µg P/l
Chloride, Sulphate, Calcium, Magnesium, Sodium, Potassium, TOC: mg/l
Aluminium, Iron, Manganese, Cadmium, Lead, Copper, Nickel and Zinc: µg/l

4 Results

In the current edition of the ICP-Waters intercomparison 88 laboratories were invited to participate in. 38 laboratories from 21 different countries accepted the invitation and they signed up in NIVA's database. When signing up, the participants were invited to fill the required information about their institution and to order the samples sets they wanted to analyse. After that, the samples were prepared and shipped to them. At the end of the program, almost all the laboratories that agreed to participate had submitted results to the Programme Centre. The participants and the numerical identity employed along the report are listed in Appendix A. In the same appendix, a table summarizing the number of laboratories that participated in the 2017 intercomparison and the represented countries can be also found.

The analytical results received from the laboratories were treated by the Youden method (2, 3). A short description of this method and the statistical treatment of the analytical data are presented in Appendix C. The purpose of this test is to evaluate the comparability of the analytical results produced by the laboratories participating in the International Cooperative Programme. The real "true value" is not known exactly for the natural water samples used in this intercomparison. Therefore, the median value -determined from the analytical results submitted by the participating laboratories after excluding outliers- was selected as the "true value" for each analytical variable. The median value is considered to be an acceptable estimate of the true value for this purpose, as long as most of the participants are using essentially the same analytical method. For certain variables, for instance pH, this may represent a problem as the different methods used may produce systematically different results (stirring, non-stirring, and equilibration of the test solution), and we cannot argue that one method is more correct than the others. Table 6 in Appendix C provides an estimate for the uncertainty of the assigned true values. This calculation is performed according to ISO 13528 (2005), "Statistical methods for use in proficiency testing by interlaboratory comparisons".

The results are illustrated in Figures 1-20, where each laboratory is represented by a small circle and an identification number. Some laboratories with strongly deviating results may be located outside the plot. The big circle in the figure, centred in the intersection of the median axes, represents a selected accuracy limit, either the general target limit of $\pm$ 20 % of the mean true values for the sample pair, or a special accuracy limit as defined in the sections below.

A summary of the results of intercomparison 1731 is presented in Tables 1 and 2. The individual results of the participants are presented in Table 4 in Appendix D, sorted by increasing identification number. More extensive statistical information is presented in the Tables 5.1 - 5.20 in the same appendix.

4.1 pH

The reported results for pH are graphically presented in the Youden graph (Figure 1), where the radius of the circle is 0.2 pH units, and shows the degree of comparability between the pH results from the participating laboratories. The values reported by the laboratories and the statistical calculations are presented in Table 2 and Table 5.1.

32 participants determined pH in the test samples A and B. 27 laboratories used a method based upon electrometry. As stated in previous intercomparisons, stirring has been observed that could have a significant influence on the results, especially in samples with lower total ion strength than

the samples used in this intercomparison (4, 5). As a result of this, the practice of establishing a “true value” based on the median value for all the reported results for pH is questionable. Whether an individual “true value” for each method would be more appropriate should therefore be discussed. In this intercomparison it was chosen the median value of all the reported results after excluding the outliers. Based upon this, 53 % of the results were acceptable, that is within the median value ± 0.2 pH units. The acceptance is comparable to that observed in the former edition (Table 1). The logarithmic operation performed over the variable strongly contributes restricting the number of acceptable data and does not mean the participants are not measuring well. Another probable reason for the differences in the reported results could be the slight differences in the analytics that the different participants employed. It is also questionable whether there could be some differences due to instability of the samples during their shipment. Stability tests performed at NIVA in previous years have demonstrated that samples are stable if stored in the dark at 4 ºC.

Noteworthy is also the presence of important systematic errors in the determination of pH as illustrated in Figure 1 by the spread of the results away from the 45° line for many laboratories in the characteristic elliptical distribution.

4.2 Conductivity

The Youden chart for conductivity results is presented in Figure 2, where the large circle represents an accuracy limit of ± 10 %, which is only half of the target accuracy limit given in the Manual (1). The values reported by the laboratories are presented in Table 2 and Table 5.2.

31 laboratories have reported results for conductivity in the current edition. 30 participants reported the use of electrometric methods. Most laboratories achieved rather good agreement between the results for this variable, 77 % of the results were within the acceptance limit of ± 10 %.

Conductivity is affected mainly by systematic errors, as it can be observed in the distribution of the results in Figure 2. It has to be pointed out that an accurate temperature control or proper temperature correction is necessary when determining this variable, as the conductivity is changing by about two percent pr. °C at room temperature.

4.3 Alkalinity

The Youden chart obtained in the determination of the alkalinity in samples A and B is illustrated in Figure 3. The statistical results are presented in Tables 2 and 5.3.

23 laboratories reported results for alkalinity. From them, 6 used Gran plot titration method, which is the suggested reference method in the manual (1), while 9 made use of end point titration. 2 participants employed end point titration to pH 5.4. 17 % of them provided results that were within the target accuracy of ± 20 %.

It is worth noting that the alkalinity value may vary significantly with the end-point pH used for the titration. In waters containing high concentrations of total inorganic carbon, the equivalence point is close to pH = 5.4. In such case, the relative error introduced by assuming a fixed end-point pH, is negligible. However, at lower alkalinities normally encountered in areas sensitive to acidification, the “total fixed end-point method” may overestimate the true alkalinity or the “equivalence” alkalinity.

The distribution of the results in the Youden's chart indicates that the analysis is affected mainly by systematic error.

4.4 Nitrate + nitrite-nitrogen

31 laboratories reported results for nitrate + nitrite-nitrogen and the results are presented in Tables 2 and 5.4. Ion chromatography is the preferred technique for the determination of this variable in the samples, as it was used by 15 participants. A decrease on the quality of the overall dataset has been observed, since just 35% of the results were considered as acceptable. The Youden plot demonstrates that the deviation in the results is mainly due to systematic error.

4.5 Chloride

28 laboratories reported results for chloride and, from them, 23 were accepted. 82% of the participants provided results that fulfilled the acceptance criteria. The results are presented in Figure 5, Table 2 and Table 5.5. The target accuracy of ± 20 % is represented by the circle in Figure 5.

Ion chromatography appears as the most widely employed technique, with 19 of the participants reporting its use. Other techniques such as photometry, capillary electrophoresis and others were employed in much lower extension. A high accuracy in the results provided by the participants was in general observed, as it might be observed in characteristic Youden plot. Just slight random error affected the analytics.

4.6 Sulphate

29 laboratories reported results for sulphate. From them 90% fulfilled the target accuracy. The results obtained for the analysis of sulphate are presented in Figure 6, Table 2 and Table 5.6.

The circle in Figure 6 represents the target accuracy of ± 20%. As in the case of chloride, most of the laboratories (19 participants) used ion chromatography as the analytical technique in their determinations of sulfate. 3 participants reported the use of ICP-AES for the determination of this variable, 3 made use of photometry and 1 potentiometry.

Due to the small number of methods other than ion chromatography, it is not possible to discuss much about differences between them, but it can be concluded that both, IC and ICP-AES provided accurate results with relative standard deviations lower than 2%. As in the case of chloride, the Youden chart demonstrates the excellent accuracy of the results provided by the participants. Just slight systematic error inside the 20% deviation from the target value was detected.

4.7 Calcium

30 laboratories reported results for calcium from which 83% fulfilled the target accuracy. This percentage is slightly lower than the observed in the last editions. The results are presented in Figure 7, Table 2 and Table 5.7. The circle in Figure 7 represents the target accuracy of ± 20%.

9 laboratories used ICP-AES and 9 ion chromatography. Flame atomic absorption spectrometry was used by 3 participants in their determination of calcium. 6 laboratories used ICP-MS. 1 participant made use of an electrophoretic technique and another determined the variable using complexometry with EDTA. The results are mainly affected by slight systematic and random error.

4.8 Magnesium

30 laboratories reported results for magnesium and 93% of the results were considered as acceptable according to the criteria of the intercomparison.

The characteristic Youden chart obtained in the current edition is presented in Figure 8. Statistical results can be found in Tables 2 and 5.8. The circle in Figure 8 represents the target accuracy of ± 20 %. 8 of the laboratories reported the use of ICP-AES, 7 employed ICP-MS and 9 ion chromatography. Flame atomic absorption spectrometry was used by 3 of the participants in their determination of this variable. 1 participant reported the use of capillary electrophoresis and 2 indicated the use of other method different than the aforementioned. There's just a slight deviation in the distribution of the results from the target and seems a binary contribution of slight random and systematic error (Figure 8).

4.9 Sodium

29 laboratories reported results for sodium. 86% of the results fulfilled the target accuracy stablished in the intercomparision. This is in agreement with the percentage of acceptance of previous editions.

The characteristics Youden chart is presented in Figure 9. Tables 2 and 5.9 summarize the statistical treatment of the data. The circle in Figure 9 represents the target accuracy of ± 20 %. In this round of the intercomparison, 7 participants analysed sodium by ICP-AES and 6 by ICP-MS. Ion chromatography techniques are nearly as extended as plasma techniques, as 9 of the participants reported the use of ion chromatography in this analytical determination. Among the flame techniques, atomic absorption is the preferred, as it was used by 4 laboratories. 1 participant reported the use of emission in flame. Just 1 laboratory reported the use of capillary electrophoresis and 1 indicated the use of other method different than the aforementioned. As in previous editions, the determination of sodium keeps a very good quality and there were no strong differences in the results obtained by the different analytical techniques. According to the distribution of the results in the Youden chart obtained in the determination of sodium, it is noticeable the overall high accuracy and exactitude of the results provided by the participants.

4.10 Potassium

29 laboratories reported results for potassium. From these results, 69% were acceptable. Regarding the analytical techniques used by the participants, the similar distribution as in the case of the analysis of sodium was evidenced. The Youden graphic obtained for the determination of potassium in this round is presented in Figure 10. Statistics results for this variable are presented in Tables 2 and 5.10. The circle in Figure 10 represents the target accuracy of ± 20%. The Youden chart points out that the deviating results are affected by systematic error and there's a higher dispersion than in the case of the determination of sodium.

4.11 Total organic carbon

21 laboratories reported results for total organic carbon. From them, 81% of the results were within the target accuracy of ± 20%. The results of the Youden test are presented in Figure 11, while the statistics can be found in Tables 2 and 5.11. The circle in Figure 11 represents the target accuracy of ± 20%. Combustion methods are used by most of the laboratories (14) while 5 reported the use of

UV/peroxodisulfate oxidation method for this determination. 2 laboratories reported the use of other method. Not significant differences were observed in the results provided by the combustion and the UV/peroxodisulfate methods. The distribution of the results in the Youden's chart demonstrates that the deviating results are mainly affected by both, random and systematic error.

4.12 Total P

19 laboratories reported results for Total P. From these 4 were accepted according (21% of total). The results of the Youden test are presented in Figure 12, where the circle represents the target accuracy of ± 20%. The statistics of the analytics are presented in Tables 2 and 5.12. In the current edition, 11 laboratories employed photometry, 4 ICP-AES and 4 other methods.
According to the distribution of the results in the Youden chart it can be stated that the deviating results are mainly affected by systematic error with slight contribution also of random error.

4.13 Aluminium

22 laboratories reported results for aluminium. From these 18 were accepted according to the target accuracy criteria (82% of total). The results of the Youden test are presented in Figure 13, where the circle represents the target accuracy of ± 20%. The statistics of the analytics are presented in Tables 2 and 5.13. In the current edition, 10 laboratories used ICP-MS and 7, ICP-AES. 3 participants reported the use of graphite furnace. 1 participant reported the use of a photometric method. From these techniques, the lowest relative standard deviation in the results was observed for the ICP-MS technique.
According to the distribution of the results in the Youden chart it can be stated that the deviating results are mainly affected by systematic error with slight contribution also of random error.

4.14 Iron

23 laboratories provided results for iron and 74 fulfilled the target accuracy criteria. The results of the Youden test are presented in Figure 14. The statistics calculations are presented in Table 2 and Table 8.14. The circle in Figure 14 represents the target accuracy of ± 20%. 9 and 10 of the laboratories used ICP-AES and ICP-MS, respectively. 3 participants reported the use of atomic absorption techniques: 2 employed GFASS and12 FAAS. 1 laboratory reported the use of a photometry-based method.
The Youden chart puts into evidence that deviating results are mainly affected by random error.

4.15 Manganese

23 participants reported results in the analysis of manganese and all of them fulfilled the acceptance criteria. The Youden chart is presented in Figure 15 and the statistical results in Tables 2 and 5.15. The circle in the figure represents the target accuracy of ± 20%.
All the participants reported the use of atomic techniques. From them, 9 and 10 participants used ICP-AES and ICP-MS, respectively, while 2 and 2 used graphite furnace atomic absorption and flame atomic absorption respectively. No relevant differences were detected in between the different techniques. The analysis is just affected by a slight systematic error, as shown in the characteristic Youden chart.

4.16 Cadmium

24 laboratories reported results for cadmium in the set of samples C and D. 92% of the results were acceptable, according to the target accuracy.

The Youden graph for cadmium is presented in Figure 16 while the statistical calculations for this variable are presented in Tables 2 and 5.16. The circle in Figure 16 represents the target accuracy of ± 20%. Plasma techniques have been the most employed, as 19 participants reported its use. From them, 13 detected mass (ICP-MS) and 6 emitted radiation (ICP-AES). The preferred method employed by the participants that used atomic absorption techniques was the graphite furnace (GFAAS). The use of this technique was reported by 4 of the participants. In the current edition, any participant reported the use of non-atomic techniques. According to the Youden chart, the deviating results seem to be affected by slight systematic error.

4.17 Lead

24 laboratories reported results for lead in samples C and D. From these, 95% were acceptable. This percentage is in line with previous intercomparisons. Youden chart is presented in Figure 17 and statistical results in the determination of this variable in Tables 2 and 5.17. The circle in Figure 17 represents the target accuracy of ± 20%. In this case, all the laboratories have reported the use of atomic techniques. Plasma techniques have been the most employed, as 19 participants have communicated the use of ICP. From them, 13 used mass detection (ICP-MS) and 6, emitted radiation (ICP-AES). The preferred method employed by the participants that used atomic absorption techniques was the graphite furnace (GFAAS). As it can be observed in the characteristic Youden chart, the results exhibit a slight random error.

4.18 Copper

22 laboratories reported results for copper in sample set C and D. From them, 95% were acceptable. Youden chart is presented in Figure 18 and statistical results in the determination of this variable in Tables 2 and 5.18. The circle in the figure represents the target accuracy of ± 20%. As it can be seen in the figure, almost all the results lied in the target accuracy stablished and the deviation in the results can be assigned mainly to random error with slight contribution of systematic error.
By analysis, almost all the participants employed atomic based techniques, being plasma the most widely used with 12 of the participants using mass detectors and 6 using emitted light. Relevant is also the contribution of atomic absorption techniques to the characterization of Cu in the samples, as 3 of the participants employed GFAAS and 1, GFAAS.

4.19 Nickel

23 laboratories reported results for nickel in samples C and D. All of them were classified as acceptable according to the target accuracy of the assay. Nickel's Youden chart is presented in Figure 19 and statistical results in Tables 2 and 5.19. The circle in the figure represents the target accuracy of ± 20%. By analysis type, it is remarkable the use of atomic based techniques. From them, plasma is the most widely used, with 19 participants. 13 employed ICP-MS while only 6 reported the use of ICP-AES. From the 4 laboratories that reported the use of atomic absorption based techniques, 3 employed graphite furnace and 1 flame atomic absorption spectroscopy. The distribution of the results in the Youden chart puts into evidence that the analysis is mainly affected by systematic error.

4.20 Zinc

23 laboratories reported results in the determination of zinc in sample set C and D. From these results, 96% fulfilled the acceptance criteria.

The Youden chart is presented in Figure 20 and statistical results in Tables 2 and 5.20. The circle in Figure 20 represents the target accuracy of ± 20 %. The elliptic distribution of the results in the Youden chart demonstrates that the determination of Zn is mainly affected by systematic error. Plasma techniques are, by far, the most widely employed by the laboratories. From them, ICP-MS demonstrated to be the most widely used, with 13 participants, followed by emission in plasma (ICP-AES) that was used by 6 of the laboratories. From the techniques based on atomic absorption spectroscopy 2 laboratories made use of the graphite furnace (GFAAS) while just 2 participants reported the use of flame atomic absorption spectroscopy (FAAS). None of the participants reported results using non-atomic techniques.

Table 2. Statistical summary for intercomparison 1731

Analytical variable and method	Sample pair	TRUE Value S. 1	TRUE Value S. 2	No. lab. Total	No. lab. Om	Median S. 1	Median S. 2	Avg/Std.av. Sample 1 (Avg)	Avg/Std.av. Sample 1 (Std)	Avg/Std.av. Sample 2 (Avg)	Avg/Std.av. Sample 2 (Std)	Rel.std.av. % S. 1	Rel.std.av. % S. 2	Relative error % S. 1	Relative error % S. 2
pH	AB	5.78	5.76	32	2	5.78	5.76	5.73	0.24	5.76	0.23	4.1	4.0	-0.9	-0.1
Electrometry				27	1	5.78	5.78	5.76	0.21	5.78	0.22	3.7	3.8	-0.4	0.4
Stirring				4	1	5.64	5.67	5.56	0.38	5.60	0.34	6.8	6.0	-3.7	-2.7
Other method				1	0			5.40		5.50				-6.6	-4.5
Conductivity	AB	2.90	2.55	31	3	2.90	2.55	2.80	0.51	2.47	0.45	18.1	18.1	-3.5	-3.1
Electrometry				30	2	2.90	2.55	2.80	0.51	2.47	0.45	18.1	18.1	-3.5	-3.1
Other method				1	1			30.00		26.00				934.5	919.6
Alkalinity	AB	0.022	0.020	23	5	0.021	0.019	0.029	0.018	0.026	0.017	62.7	64.3	33.1	32.3
End point titration				9	2	0.037	0.035	0.037	0.015	0.034	0.017	41.7	48.9	68.3	72.2
Gran plot titration				6	0	0.021	0.018	0.025	0.011	0.020	0.004	45.6	20.2	13.6	-0.7
End point				3	1			0.045		0.042				104.8	111.6
End point 5.4				2	0			0.005		0.008				-78.0	-62.0
Other method				2	2			1.654		1.300				7418.2	6465.7
End point 5.6				1	0			0.018		0.016				-18.2	-19.2
Nitrate+ nitrite; N	AB	66	61	26	1	66	61	72	54	67	51	75.3	76.8	9.6	9.3
Ion chromatography				14	1	66	61	72	56	65	47	77.1	72.1	9.1	6.0
Autoanalyzer				3	0	60	50	42	36	37	32	86.8	87.9	-37.0	-39.4
Flow injection anal.				2	0			66		62				0.4	1.6
Photometry				2	0			98		107				47.7	75.4
Photometry				2	0			90		100				36.4	63.9
Cap. electrophoresis				1	0			0		0				-100.0	-100.0
Hydrazine				1	0			100		87				52.1	42.8
Other method				1	0			140		90				112.1	47.5
Chloride	AB	2.0	1.8	28	1	2.0	1.8	267.3	661.5	226.8	566.6	247.5	249.8	13266.4	12715.2
Ion chromatography				19	0	2.0	1.8	206.1	611.4	182.3	540.8	296.7	296.7	10204.6	10198.7
Other method				5	0	2.2	1.9	415.7	924.8	347.5	772.8	222.5	222.4	20682.9	19535.2
AA				1	0			1.8		1.4				-10.0	-20.3
Cap. electrophoresis				1	0			2.0		1.8				0.0	1.7
Photometry				1	0			1220.0		920.0				60900.0	51877.4
Potentiometry				1	1			7800.0		7090.0				389900.0	400465.0
Sulphate	AB	5.62	5.00	29	3	5.63	5.00	5.68	0.23	5.01	0.20	4.0	4.1	1.0	0.1
Ion chromatography				21	1	5.58	4.99	5.62	0.14	4.97	0.18	2.6	3.6	0.0	-0.7
ICP-AES				3	0	5.86	5.15	5.99	0.44	5.17	0.36	7.4	7.0	6.7	3.4
Photometry				3	1			5.88		5.18				4.7	3.6
Cap. electrophoresis				1	0			5.50		5.00				-2.1	0.0
Gravimetry				1	1			10.56		4.31				87.9	-13.8
Calcium	AB	2.40	2.09	30	0	2.40	2.09	2.42	0.32	2.12	0.24	13.4	11.3	1.0	1.6
ICP-AES				9	0	2.40	2.11	2.46	0.23	2.15	0.16	9.3	7.4	2.4	2.9
Ion chromatography				9	0	2.43	2.14	2.43	0.39	2.15	0.28	16.0	13.1	1.5	3.0
ICP-MS				6	0	2.38	2.09	2.38	0.26	2.07	0.12	10.7	5.9	-0.7	-0.8
FAAS				3	0	2.37	2.07	2.40	0.06	2.07	0.02	2.7	0.7	-0.1	-1.1
Cap. Electrophoresis				1	0			1.83		1.66				-23.7	-20.6
EDTA				1	0			3.21		2.80				33.8	34.0
Other method				1	0			2.16		1.89				-10.0	-9.6

Analytical variable and method	Sample	TRUE Value		No. lab.		Median		Avg/Std.av.		Avg/Std.av.		Rel.std.av. %		Relative error %	
	pair	S. 1	S. 2	Total	Om	S. 1	S. 2	Sample 1		Sample 2		S. 1	S. 2	S. 1	S. 2
Magnesium	AB	0.37	0.32	30	2	0.37	0.32	0.37	0.02	0.32	0.01	4.5	3.8	0.1	0.7
Ion chromatography				9	1	0.37	0.33	0.37	0.02	0.32	0.01	4.6	4.5	1.0	-0.1
ICP-AES				8	0	0.37	0.33	0.37	0.02	0.33	0.01	4.7	4.0	0.8	3.1
ICP-MS				7	0	0.36	0.32	0.37	0.02	0.32	0.01	5.7	2.6	-1.0	-0.4
FAAS				3	0	0.36	0.32	0.36	0.01	0.32	0.01	1.6	1.8	-2.0	-0.8
Other method				2	1			0.36		0.31				-2.4	-3.4
Cap. Electrophoresis				1	0			0.38		0.33				3.0	2.8
Sodium	AB	1.63	1.44	29	2	1.62	1.44	1.65	0.15	1.46	0.12	9.0	8.0	1.8	1.4
Ion chromatography				9	0	1.64	1.46	1.69	0.13	1.51	0.13	8.0	8.4	4.0	5.2
ICP-AES				7	1	1.58	1.40	1.60	0.08	1.41	0.07	5.2	4.7	-1.7	-1.8
ICP-MS				6	1	1.61	1.44	1.71	0.27	1.47	0.17	16.1	11.7	5.1	2.2
FAAS				4	0	1.60	1.42	1.60	0.09	1.42	0.03	5.7	2.3	-1.8	-1.6
AES				1	0			1.72		1.53				5.8	6.3
Cap. Electrophoresis				1	0			1.54		1.28				-5.2	-11.1
Other method				1	0			1.68		1.47				3.4	2.1
Potassium	AB	0.23	0.21	29	4	0.23	0.21	0.24	0.02	0.21	0.02	9.6	8.9	3.0	0.2
Ion chromatography				9	2	0.23	0.21	0.23	0.02	0.20	0.02	6.7	7.9	-1.9	-4.4
ICP-AES				7	0	0.24	0.21	0.24	0.03	0.21	0.02	11.0	10.4	1.0	-0.2
ICP-MS				6	0	0.23	0.21	0.24	0.02	0.21	0.01	7.4	6.5	2.2	-1.3
FAAS				4	2			0.28		0.23				19.7	11.0
AES				1	0			0.28		0.24				20.2	14.3
Cap. Electrophoresis				1	0			0.23		0.22				-0.4	4.8
Other method				1	0			0.25		0.22				7.3	4.8
Total Organic Sarbon	AB	15.73	14.30	21	2	15.87	14.30	15.30	1.96	13.96	1.53	12.8	11.0	-2.7	-2.4
Combustion				14	1	15.87	14.29	15.46	1.83	13.79	1.65	11.8	12.0	-1.7	-3.5
UV/peroxodisulphate				5	0	16.40	15.11	15.74	1.53	14.77	0.74	9.7	5.0	0.1	3.3
Other method				2	1			11.00		12.00				-30.1	-16.1
Total Phosphorous	AB	11.50	10.00	19	1	11.50	10.00	12.04	5.47	10.49	4.77	45.4	45.5	4.7	4.9
Photometry				11	0	11.30	10.40	12.21	3.62	11.27	3.60	29.6	31.9	6.2	12.7
ICP-AES				4	1	15.00	10.00	15.13	7.03	12.61	6.91	46.5	54.8	31.6	26.1
Other method				4	0	10.00	7.50	9.25	8.54	6.75	5.37	92.2	79.6	-19.5	-32.5
Aluminium	CD	178	157	22	1	178	157	176	18	157	16	10.4	10.2	-1.4	0.0
ICP-MS				10	0	178	157	178	9	158	6	5.0	3.9	0.0	0.3
ICP-AES				7	0	172	156	177	23	158	21	12.9	13.2	-0.8	0.7
GFAAS				3	0	182	169	164	34	153	33	21.0	21.6	-8.1	-2.5
Photometry				2	1			180		157				1.1	0.0
Iron	CD	81.75	73.05	23	1	81.75	73.05	81.46	6.51	74.13	10.11	8.0	13.6	-0.4	1.5
ICP-MS				10	0	81.76	73.55	80.43	6.60	73.96	11.03	8.2	14.9	-1.6	1.2
ICP-AES				9	0	81.70	71.90	80.65	4.08	73.27	10.73	5.1	14.6	-1.3	0.3
GFAAS				2	0			81.00		73.45				-0.9	0.5
FAAS				1	1			110.00		80.00				34.6	9.5
Photometry				1	0			100.00		85.00				22.3	16.4

Analytical variable and method	Sample	TRUE Value		No. lab.		Median		Avg/Std.av.		Avg/Std.av.		Rel.std.av. %		Relative error %	
	pair	S. 1	S. 2	Total	Om	S. 1	S. 2	Sample 1		Sample 2		S. 1	S. 2	S. 1	S. 2
Cadmium	CD	9.68	8.59	24	2	9.68	8.59	9.61	0.53	8.48	0.46	5.5	5.4	-0.7	-1.3
ICP-MS				13	1	9.68	8.62	9.72	0.43	8.61	0.46	4.5	5.3	0.4	0.2
ICP-AES				6	0	9.35	8.33	9.32	0.57	8.30	0.51	6.1	6.1	-3.7	-3.4
GFAAS				4	0	9.58	8.26	9.73	0.70	8.37	0.38	7.2	4.5	0.5	-2.6
Manganese	CD	48.90	43.00	23	0	48.90	43.00	48.85	1.89	43.00	1.72	3.9	4.0	-0.1	0.0
ICP-MS				10	0	48.50	42.95	48.17	1.03	42.63	1.26	2.1	3.0	-1.5	-0.9
ICP-AES				9	0	49.40	43.00	49.09	2.05	43.30	2.15	4.2	5.0	0.4	0.7
FAAS				2	0			48.96		42.05				0.1	-2.2
GFAAS				2	0			51.10		44.50				4.5	3.5
FAAS				1	1			5.95		5.69				-38.5	-33.8
Lead	CD	7.82	6.82	24	2	7.83	6.86	7.96	0.61	6.93	0.55	7.6	8.0	1.8	1.7
ICP-MS				13	0	8.06	7.02	8.22	0.56	7.09	0.63	6.8	8.9	5.1	4.0
ICP-AES				6	0	7.60	6.53	7.46	0.35	6.59	0.32	4.7	4.8	-4.7	-3.4
GFAAS				4	1	7.51	6.94	7.89	0.71	6.95	0.15	8.9	2.2	0.9	1.9
FAAS				1	1			2.87		2.26				-63.3	-66.8
Copper	CD	29.63	26.95	22	0	29.63	26.95	29.78	1.56	26.70	1.39	5.2	5.2	0.5	-0.9
ICP-MS				12	0	29.78	26.95	29.95	1.09	26.91	0.81	3.6	3.0	1.1	-0.1
ICP-AES				6	0	29.75	26.60	29.24	2.09	26.23	1.90	7.2	7.3	-1.3	-2.7
GFAAS				3	0	29.70	27.10	30.53	2.36	27.53	1.79	7.7	6.5	3.0	2.2
FAAS				1	0			28.81		24.53				-2.8	-9.0
Nickel	CD	14.29	12.74	23	0	14.29	12.74	14.16	0.73	12.59	0.63	5.2	5.0	-0.9	-1.1
ICP-MS				13	0	14.29	12.80	14.27	0.63	12.62	0.63	4.4	5.0	-0.1	-0.9
ICP-AES				6	0	14.06	12.34	14.12	0.79	12.51	0.69	5.6	5.5	-1.2	-1.8
GFAAS				3	0	14.50	12.90	13.80	1.30	12.60	0.89	9.4	7.1	-3.4	-1.1
FAAS				1	0			13.97		12.67				-2.3	-0.6
Zinc	CD	19.68	18.42	23	1	19.68	18.42	19.71	0.88	18.50	0.91	4.5	4.9	0.2	0.4
ICP-MS				13	0	19.67	18.68	19.60	0.83	18.64	0.89	4.2	4.8	-0.4	1.2
ICP-AES				6	0	19.49	18.25	19.61	0.78	18.19	0.70	4.0	3.9	-0.4	-1.3
FAAS				2	1			21.80		20.10				10.8	9.1
GFAAS				2	0			19.70		17.70				0.1	-3.9

*Om.: Sample pair omitted from the calculations

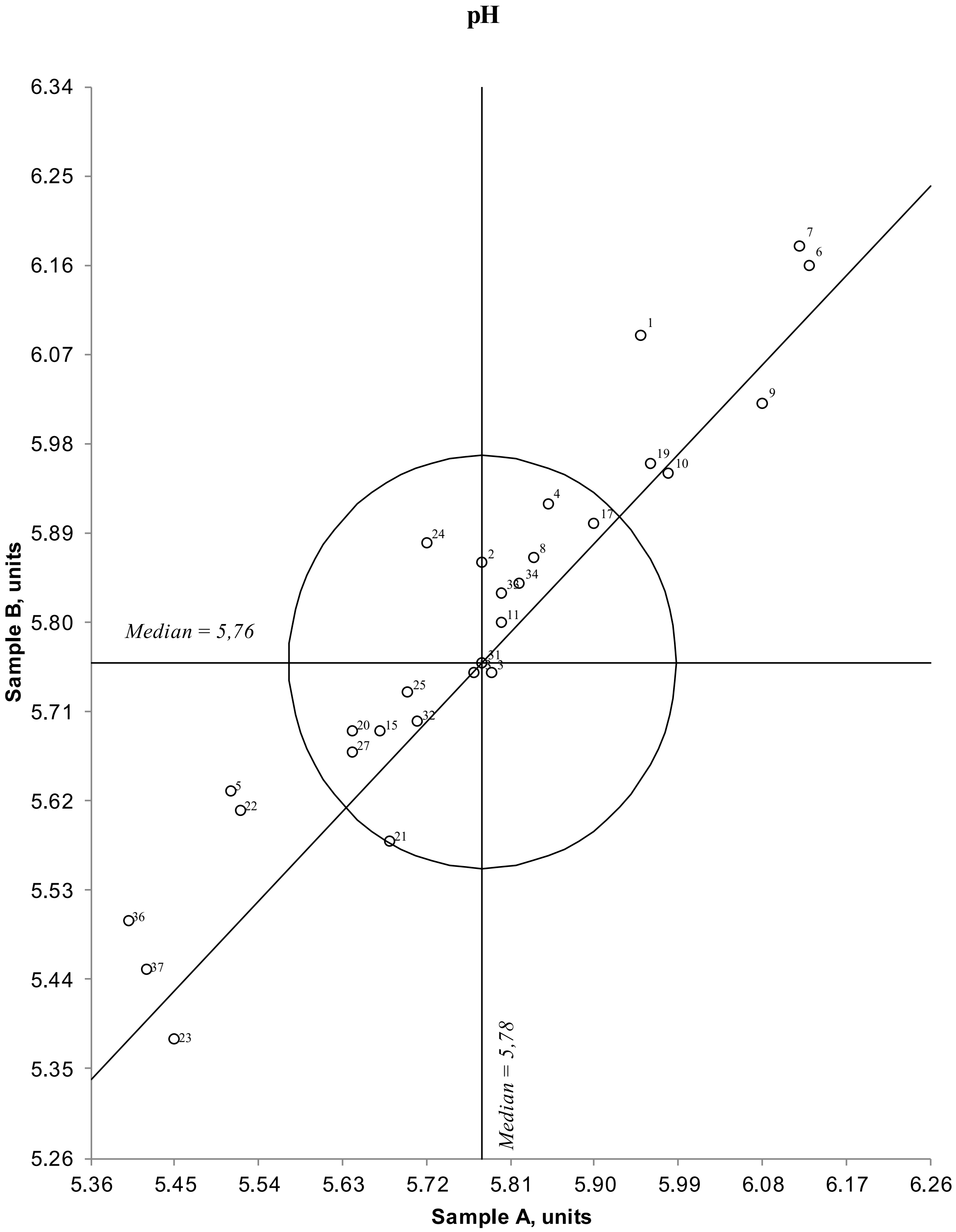

Figure 1. Youden diagram for pH, sample pair AB
Acceptable limit, given by circle, is 3,6 %

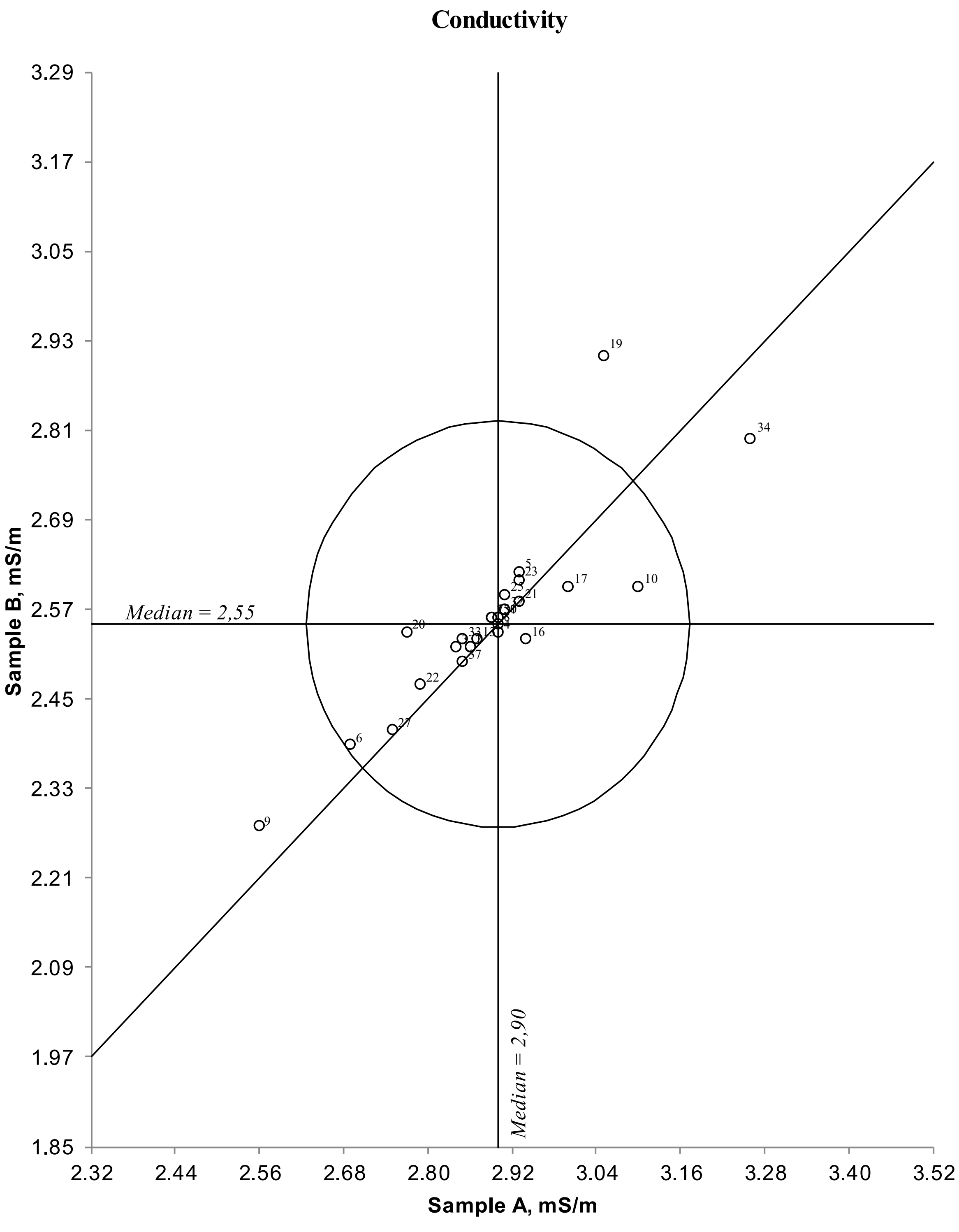

Figure 2. Youden diagram for conductivity, sample pair AB
Acceptable limit, given by circle, is 10 %

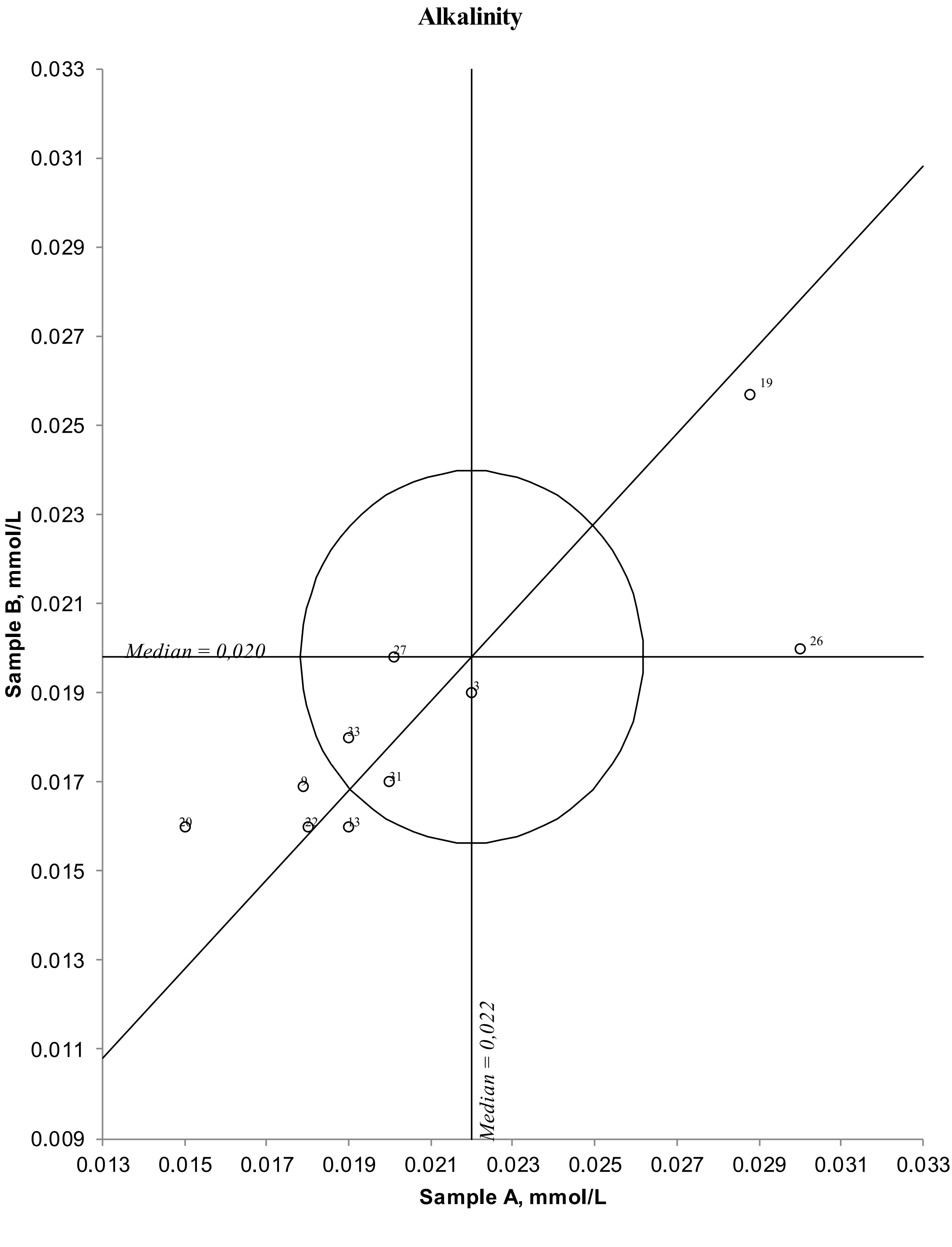

Figure 3. Youden diagram for alkalinity, sample pair AB
Acceptable limit, given by circle, is 20 %

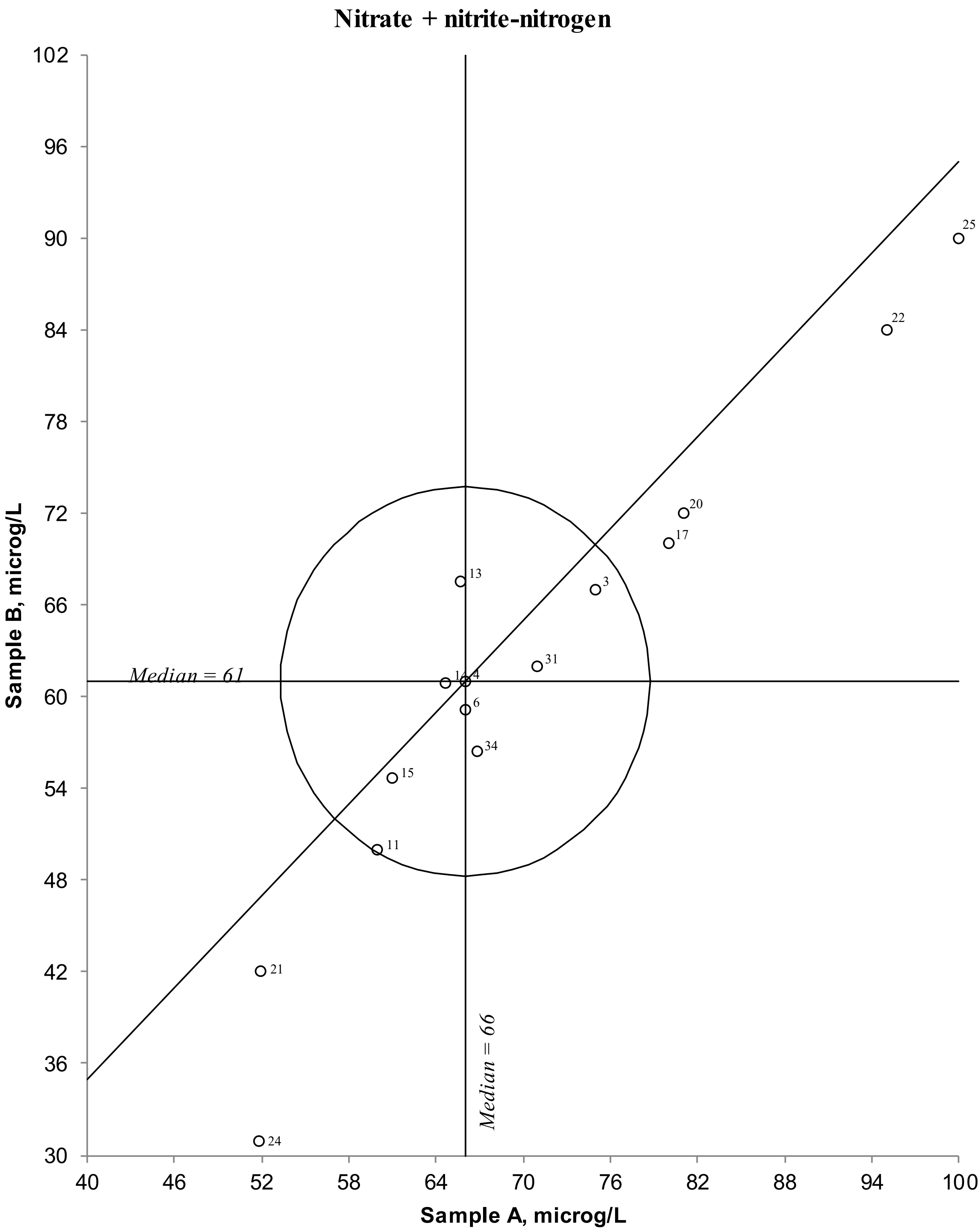

Figure 4. Youden diagram for nitrate + nitrite-nitrogen, sample pair AB
Acceptable limit, given by circle, is 20 %

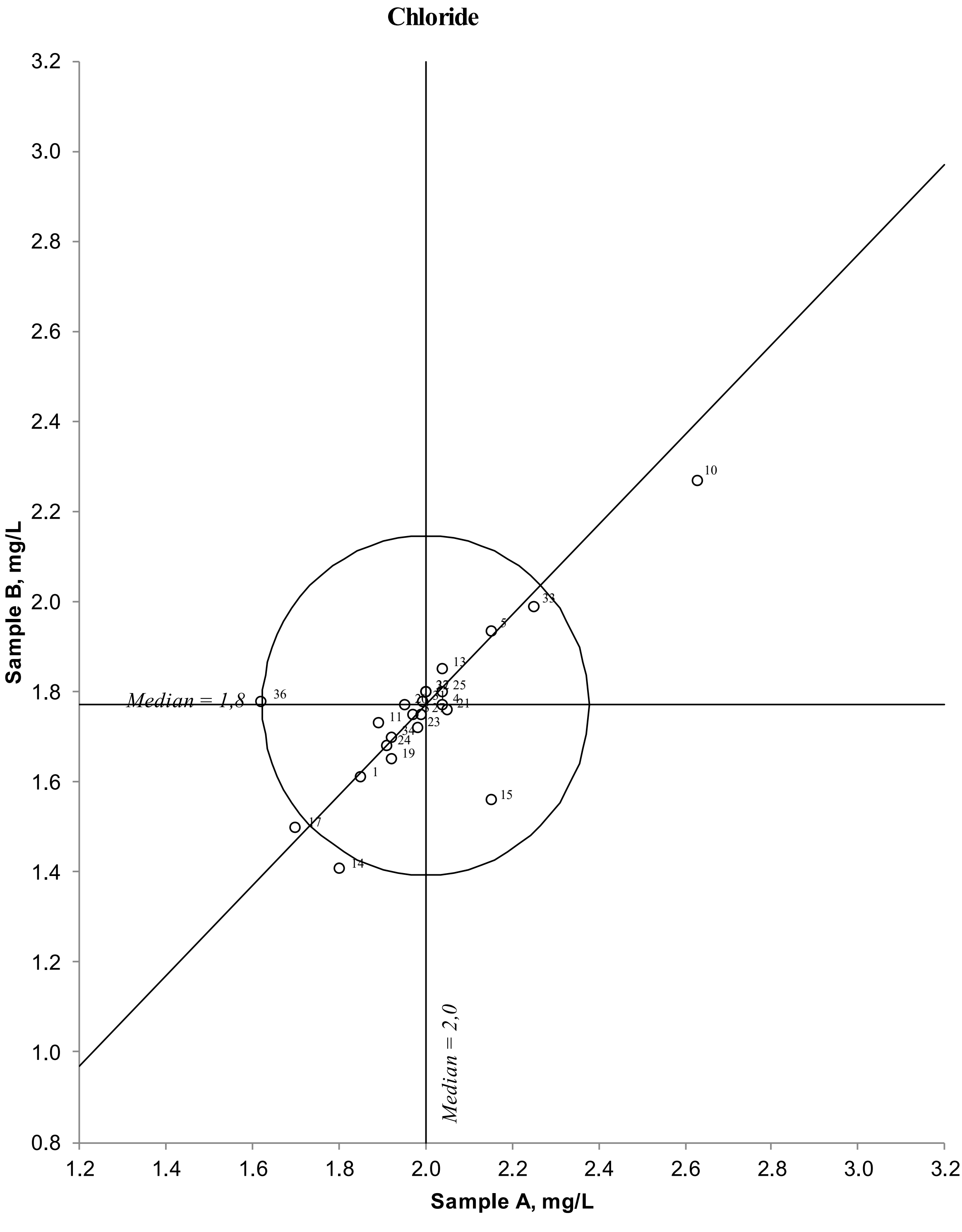

Figure 5. Youden diagram for chloride, sample pair AB
Acceptable limit, given by circle, is 20 %

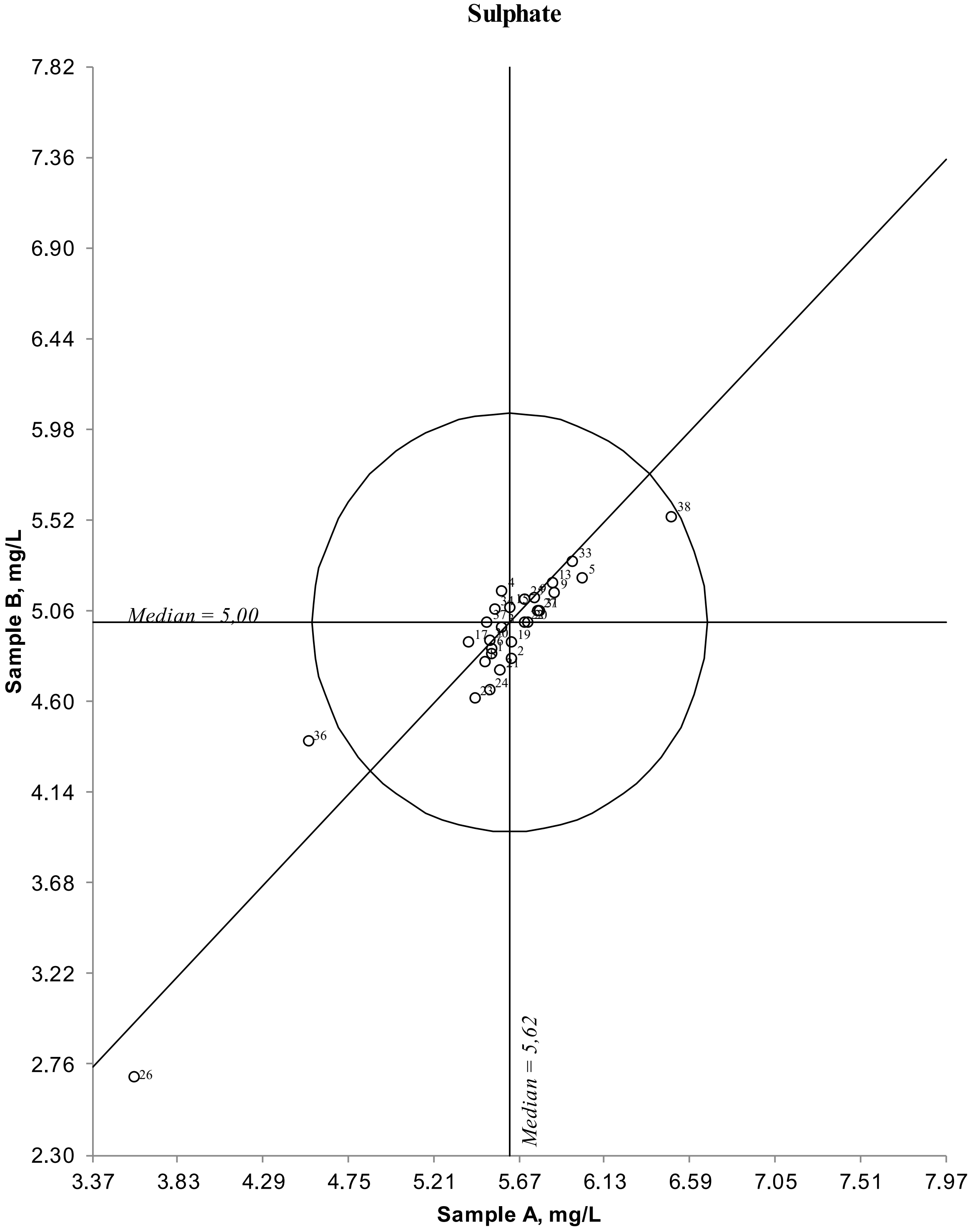

Figure 6. Youden diagram for sulphate, sample pair AB
Acceptable limit, given by circle, is 20 %

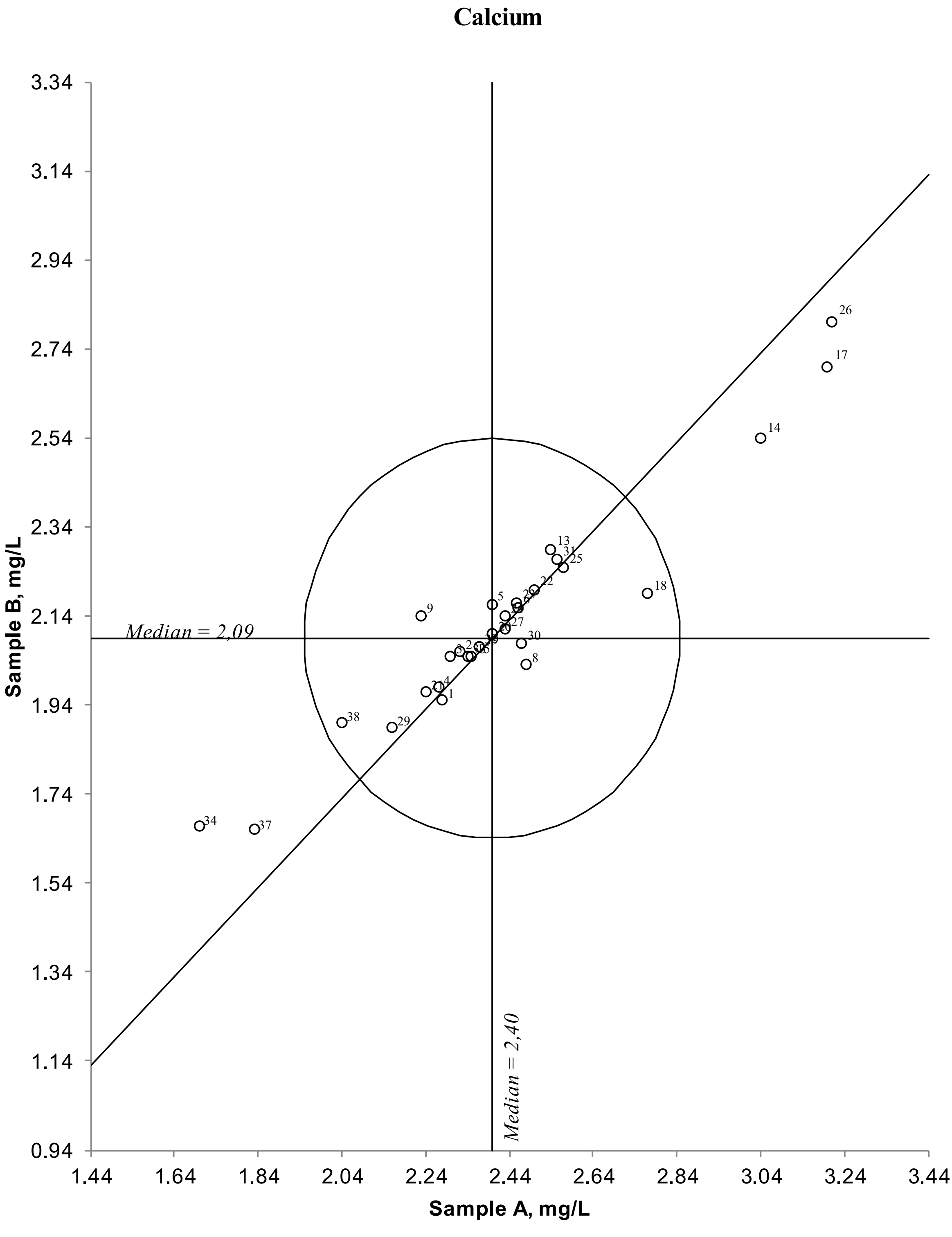

Figure 7. Youden diagram for calcium, sample pair AB
Acceptable limit, given by circle, is 20 %

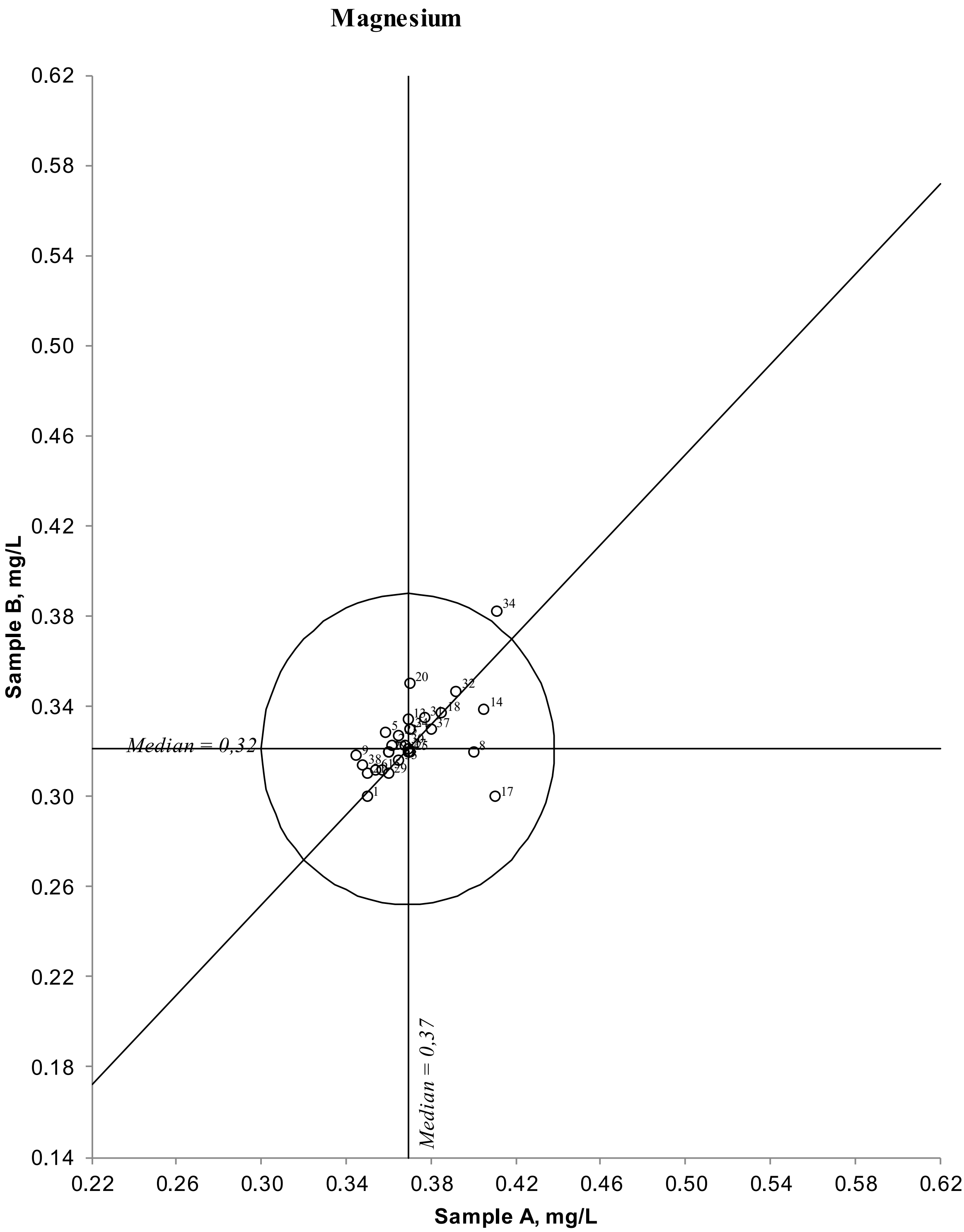

Figure 8. Youden diagram for magnesium, sample pair AB
Acceptable limit, given by circle, is 20 %

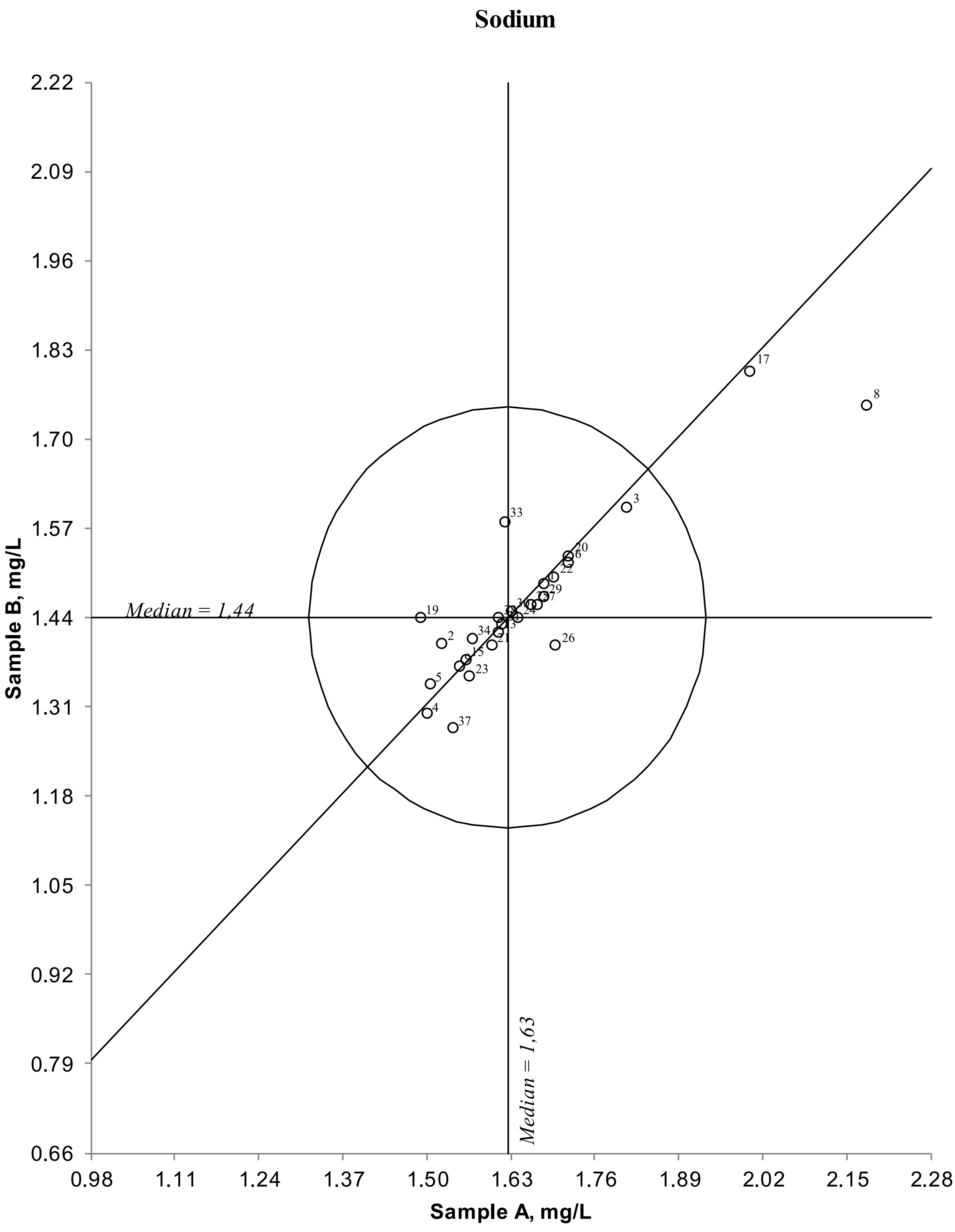

Figure 9. Youden diagram for sodium, sample pair AB
Acceptable limit, given by circle, is 20 %

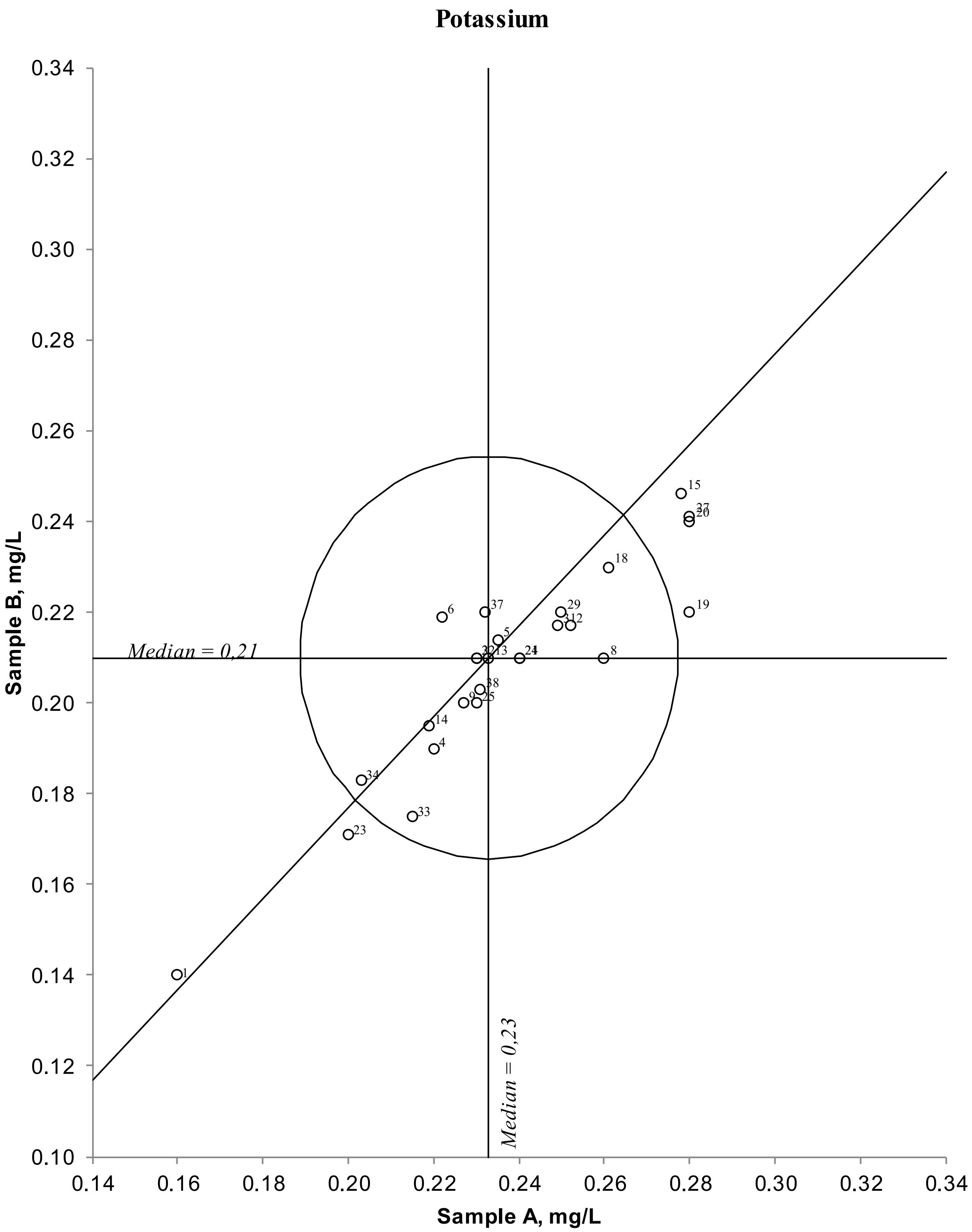

Figure 10. Youden diagram for potassium, sample pair AB
Acceptable limit, given by circle, is 20 %

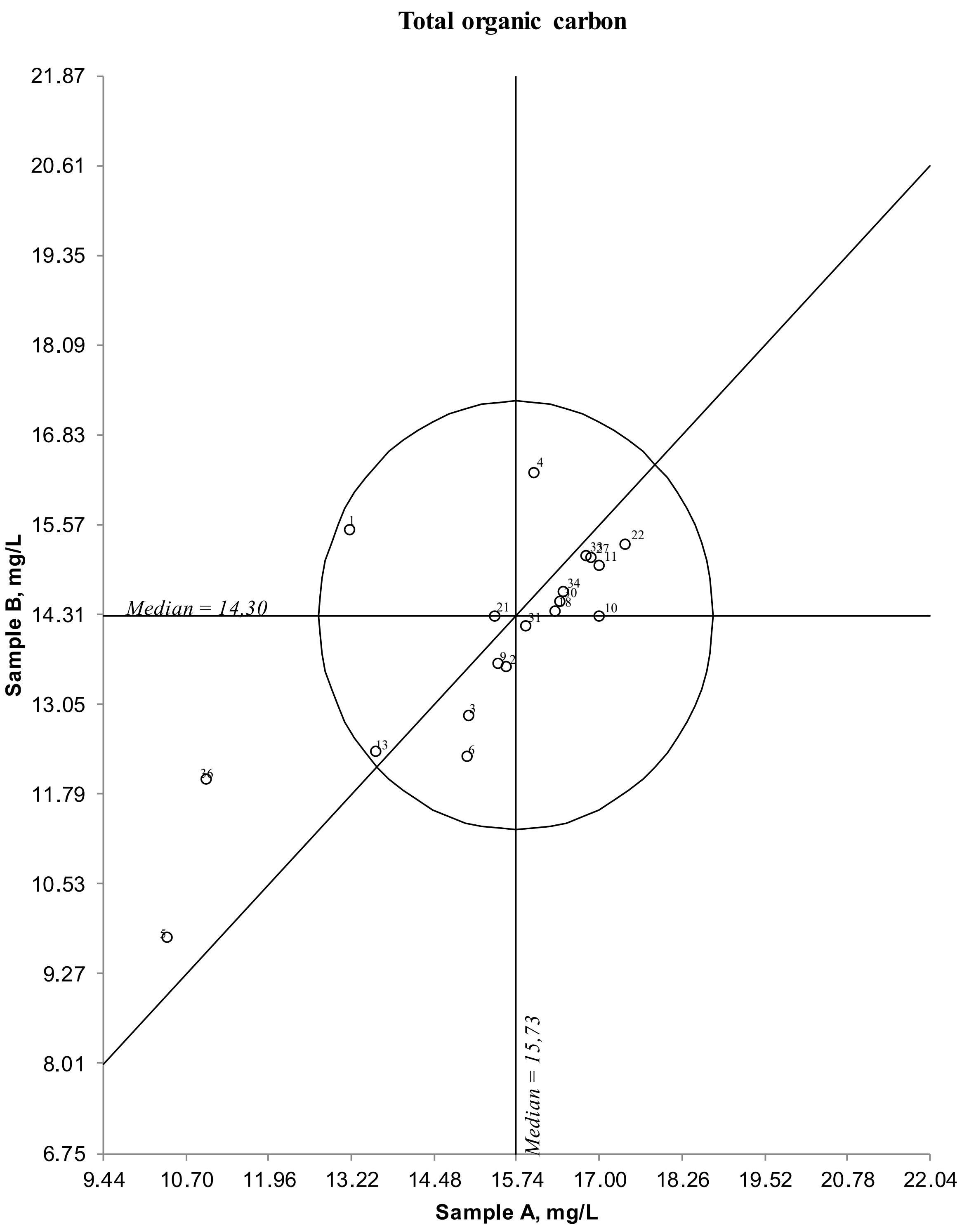

Figure 11. Youden diagram for total organic carbon, sample pair AB
Acceptable limit, given by circle, is 20 %

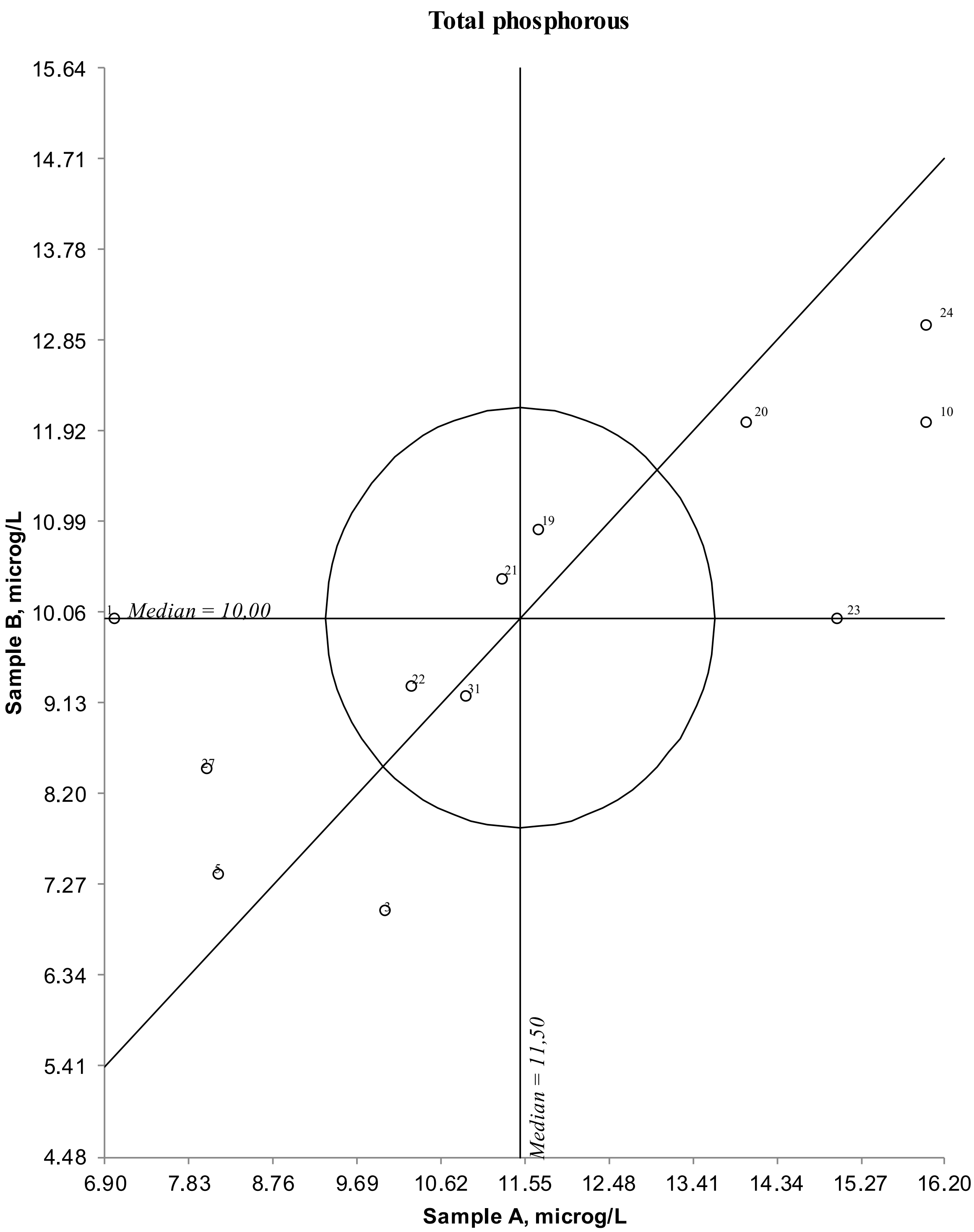

Figure 12. Youden diagram for total phosphorous, sample pair AB
Acceptable limit, given by circle, is 20 %

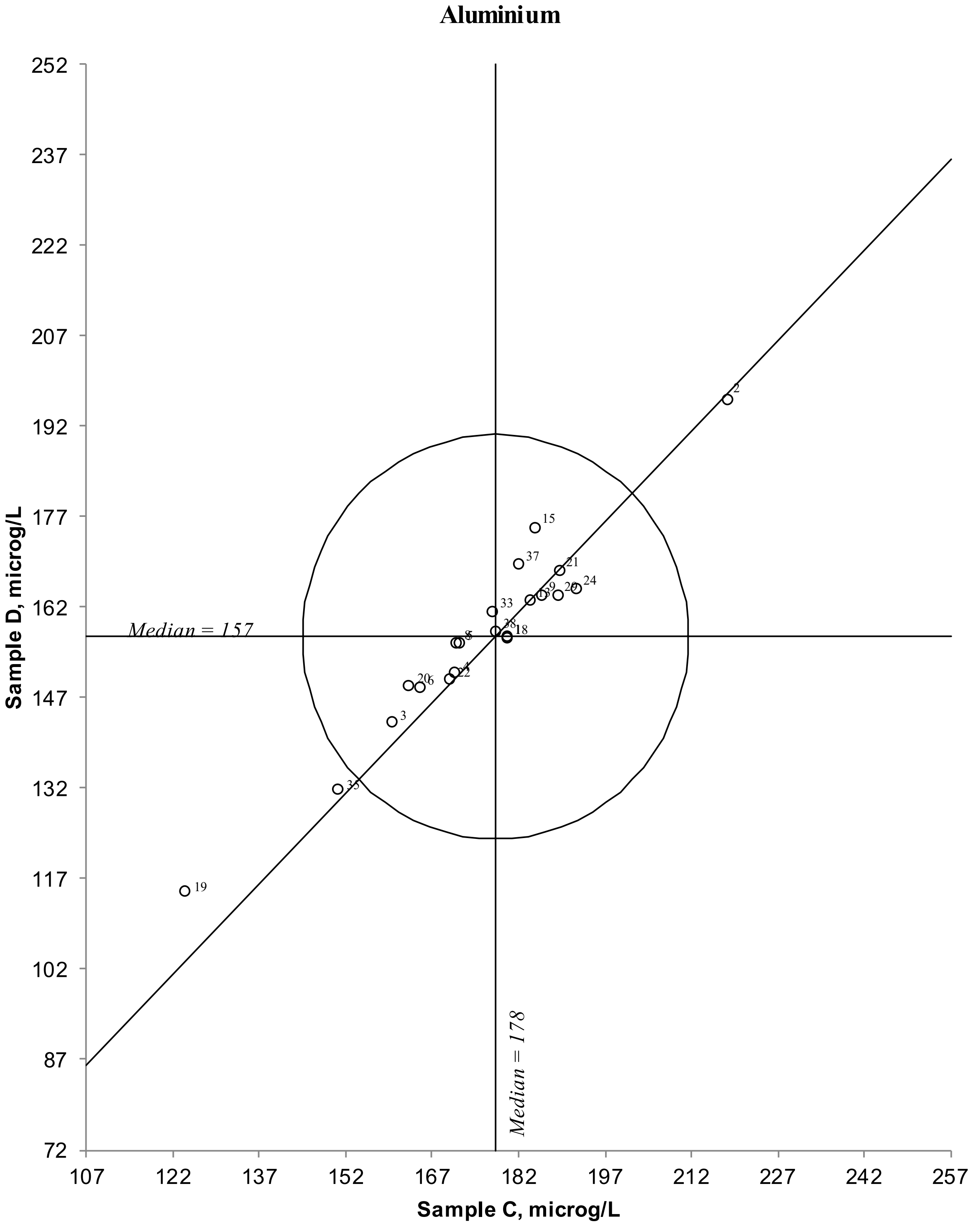

Figure 13. Youden diagram for aluminium, sample pair CD
Acceptable limit, given by circle, is 20 %

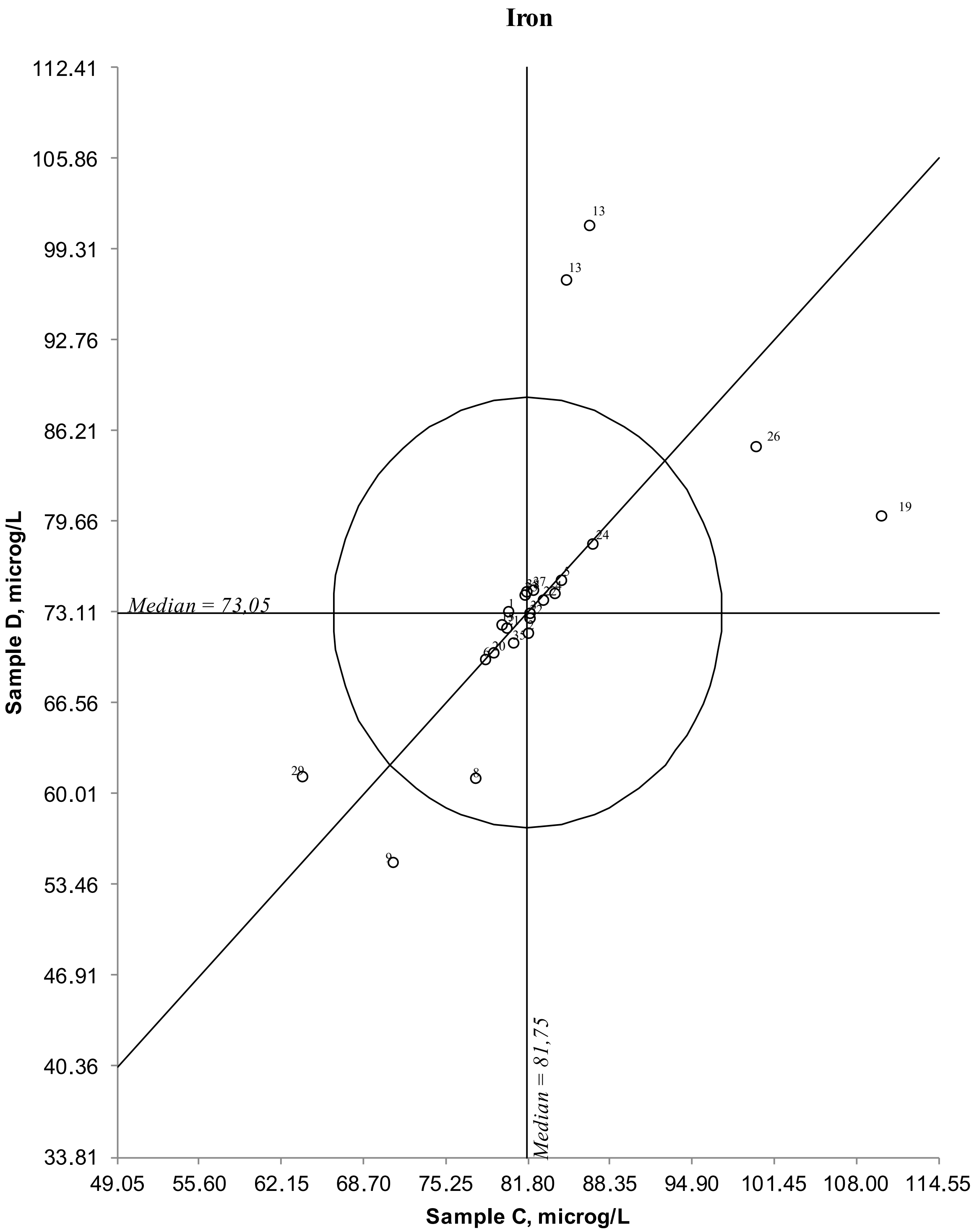

Figure 14. Youden diagram for iron, sample pair CD
Acceptable limit, given by circle, is 20 %

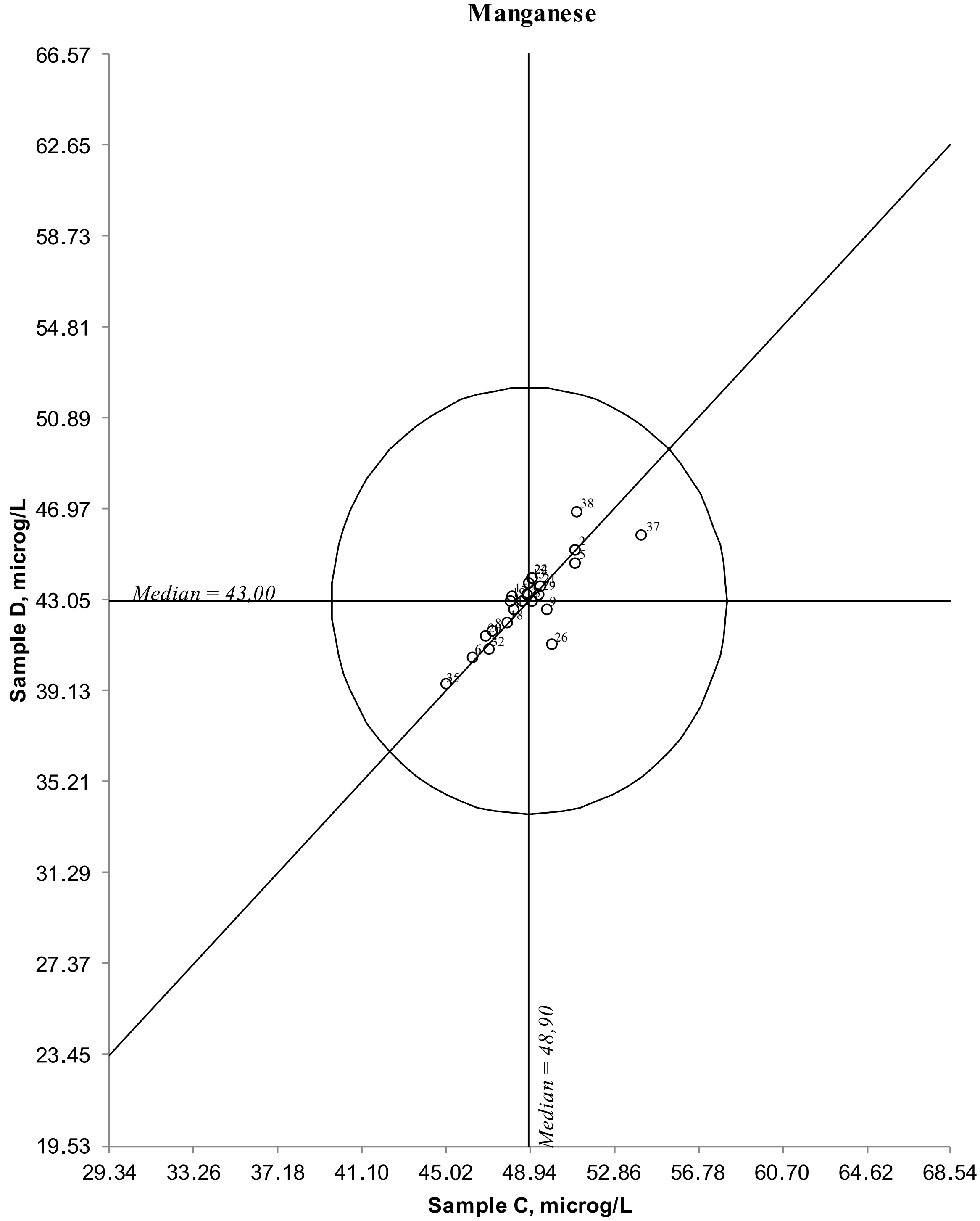

Figure 15. Youden diagram for manganese, sample pair CD
Acceptable limit, given by circle, is 20 %

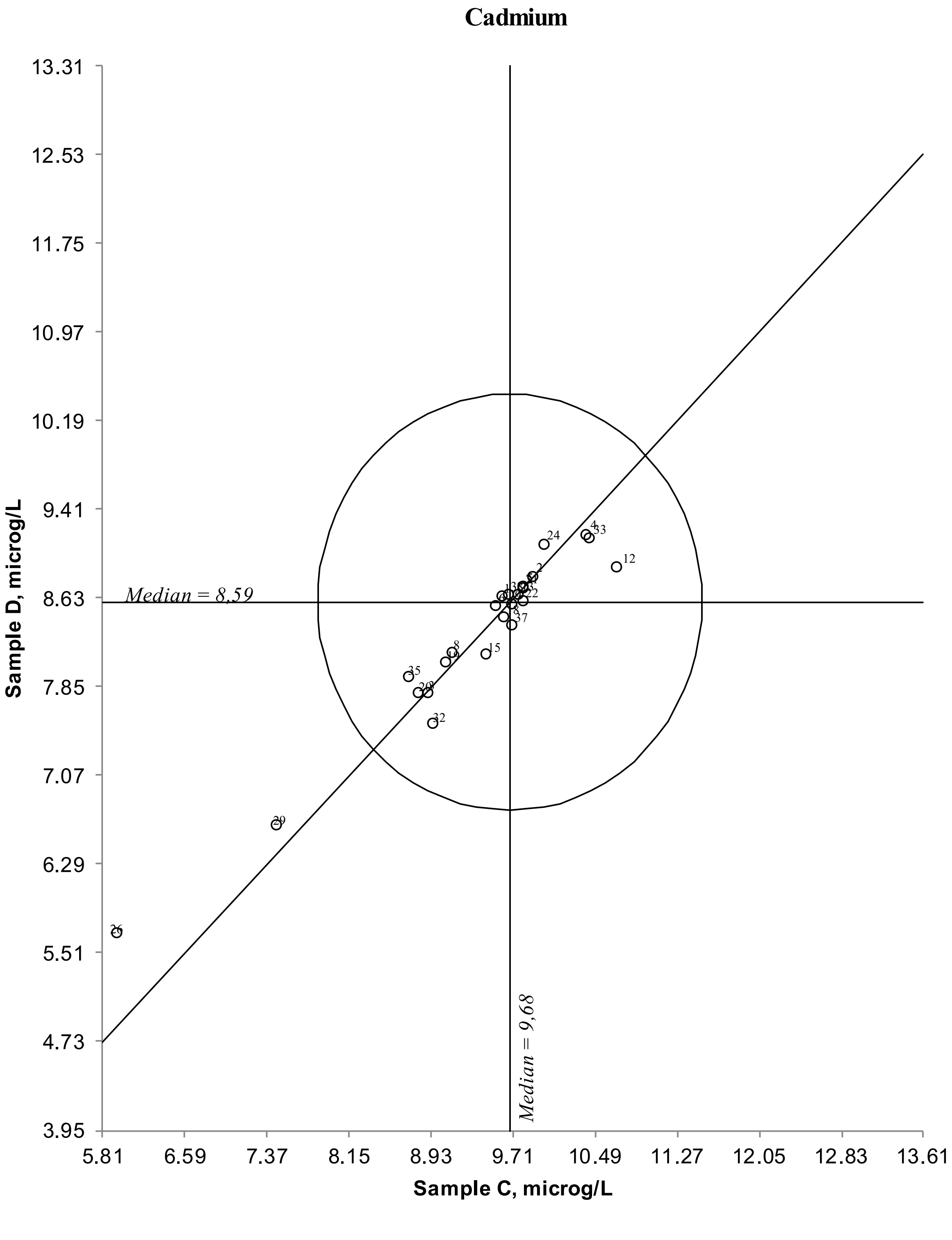

Figure 16. Youden diagram for cadmium, sample pair CD
Acceptable limit, given by circle, is 20 %

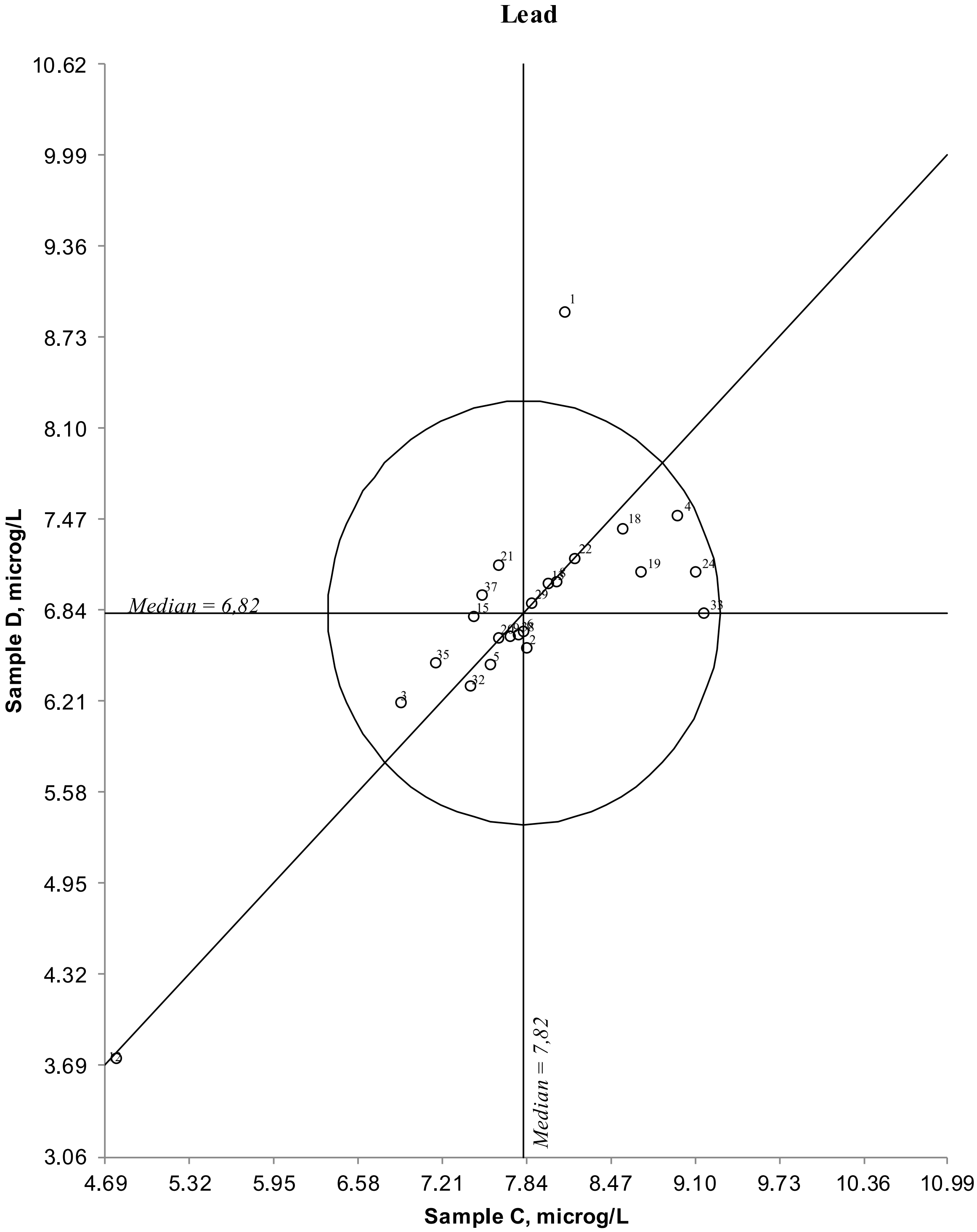

Figure 17. Youden diagram for lead, sample pair CD
Acceptable limit, given by circle, is 20 %

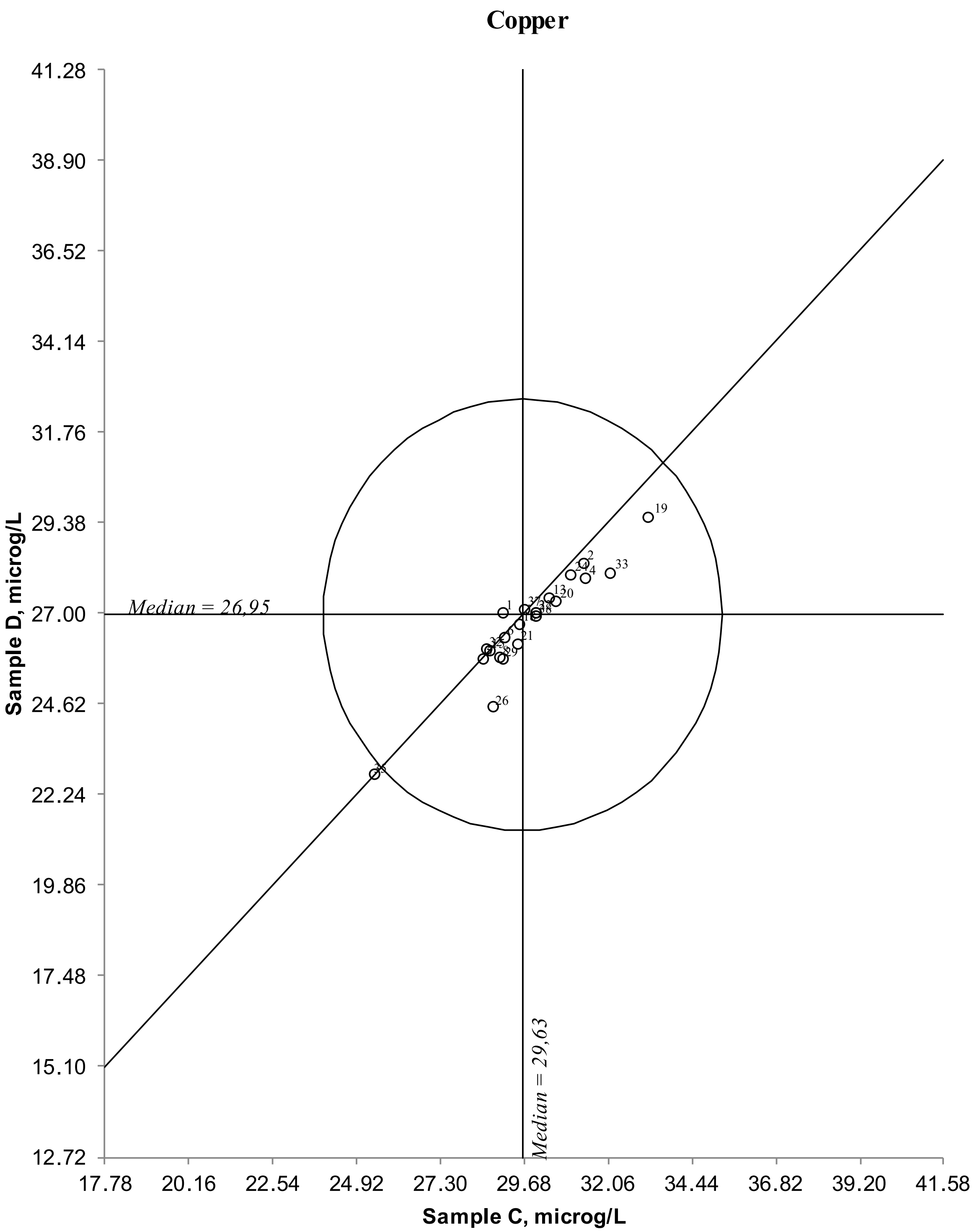

Figure 18. Youden diagram for copper, sample pair CD
Acceptable limit, given by circle, is 20 %

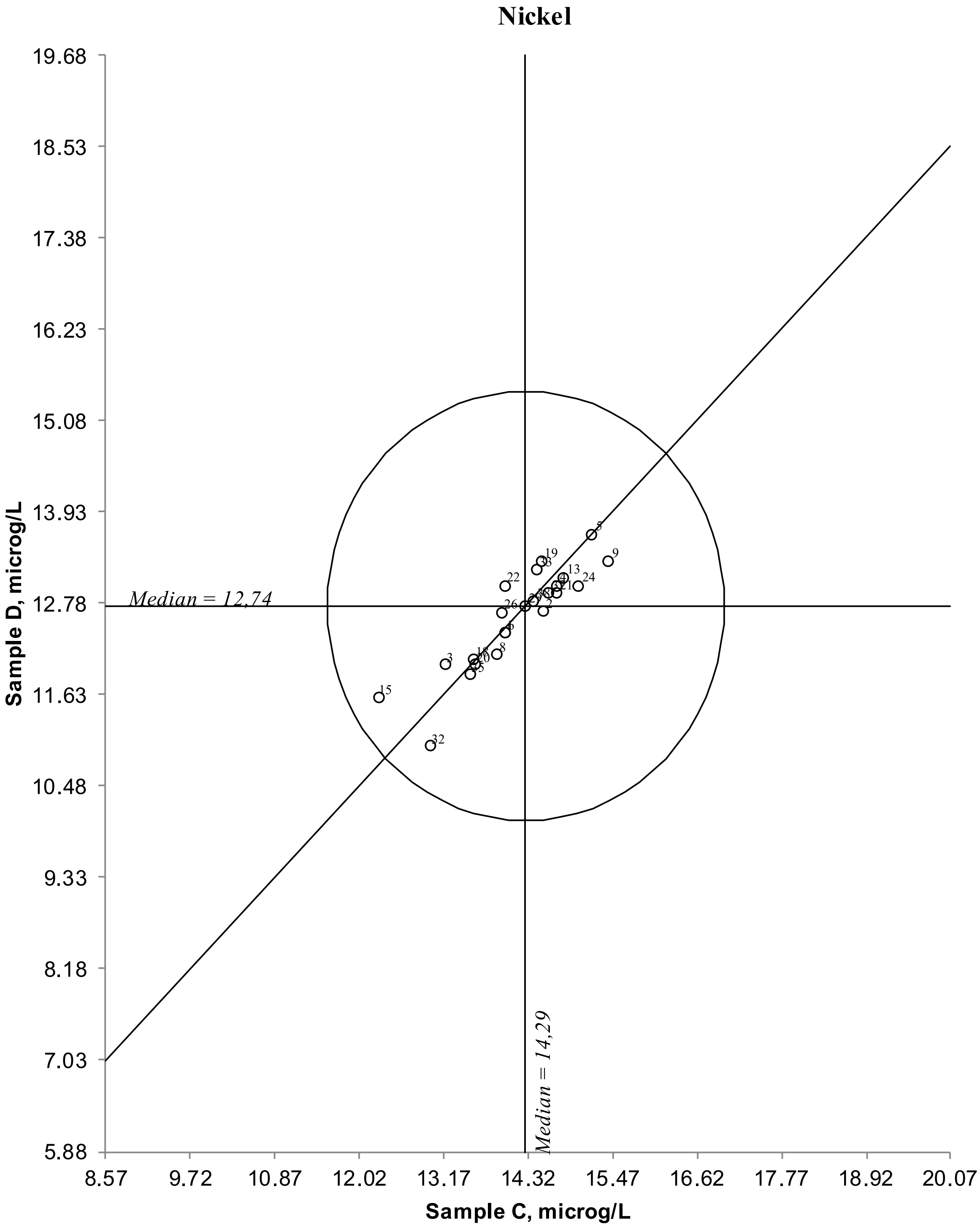

Figure 19. Youden diagram for nickel, sample pair CD
Acceptable limit, given by circle, is 20 %

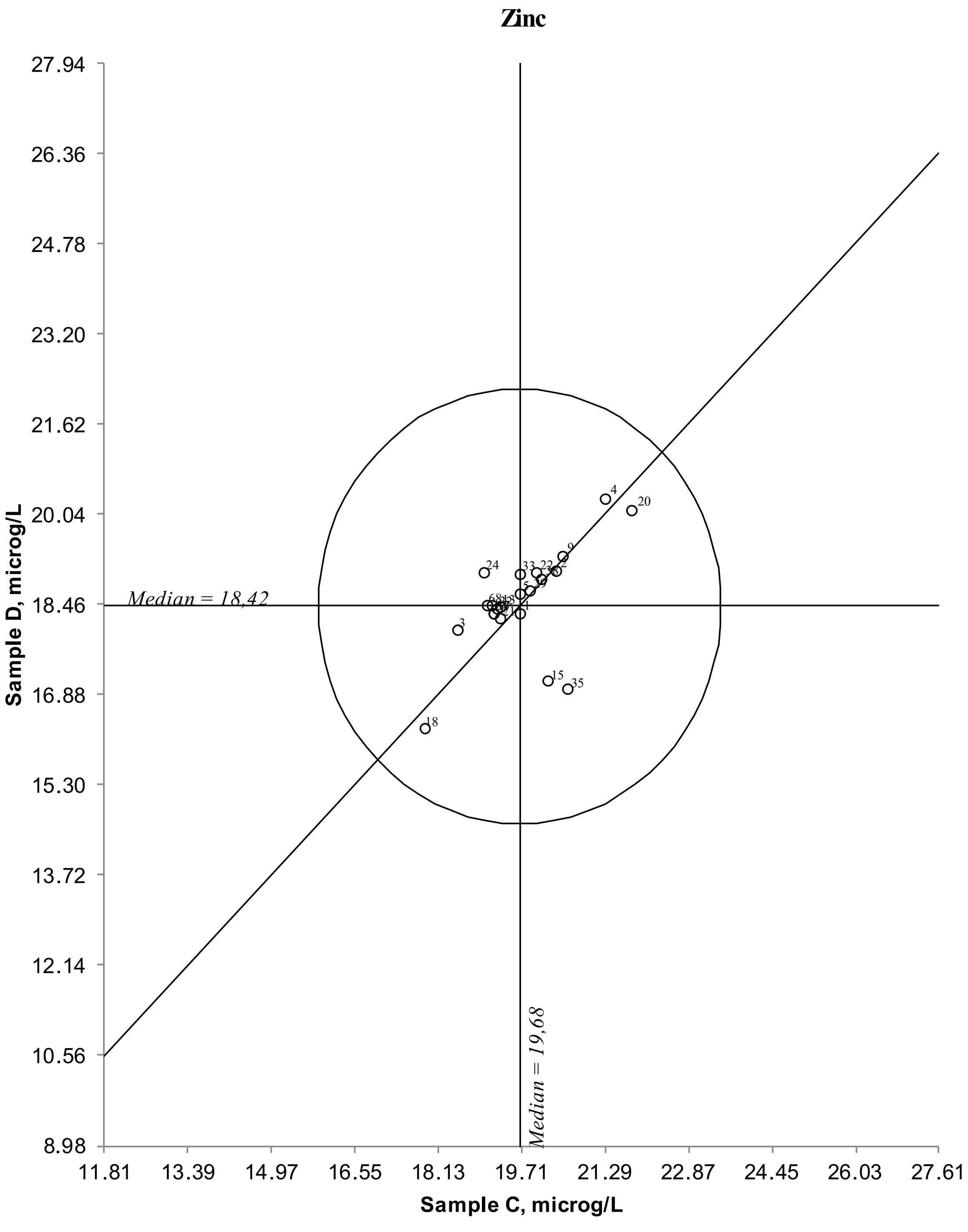

Figure 20. Youden diagram for zinc, sample pair CD
Acceptable limit, given by circle, is 20 %

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4. Hindar, A.: The Effect of Stirring on pH Readings in Solutions of Low and High Ionic Strength Measured with Electrodes of Different Condition. Vatten 1984, 40, pp 312 - 19 (in Norwegian).

5. Galloway, J.N., Cosby, B.T., Likens, G.E.: Acid Precipitation: Measurement of pH and Alkalinity. Limnol. Oceanogr. 1979, 24, 1161.

6. ISO 13528 (2005): Statistical methods for use in proficiency testing by interlaboratory comparisons.

Appendix A.

The participating laboratories

No	Laboratory	Town	Country
1	Norway	Norwegian Institute for Water Research	Gaustadalléen, 21, 0349, Oslo
2	Germany	Büsgen-Institute - Soil Science of Temperate Ecosystems	D-37077 Goettingen Buesgenweg 2
3	Italy	CNR Istituto Studio degli Ecosistemi	Largo Tonolli 50 I-28922 VERBANIA Pallanza
4	Germany	Bayerisches Landesamt fuer Umwelt	Ref 71 Bürgerm-Ulrich-Str. 160 D-86179 Augsburg
5	Russian Federation	Institute of Biology Komi SC UB RAS	Kommunisticheskaya st.,28, Syktyvkar, 167982, Russia
6	Germany	Bayerische Landesanstalt fuer Wald und Forstwirtschaft Abteilung 2 - Boden und Klima	Hans-Carl-von-Carlowitz-Platz 1 D-85354 Freising
7	Poland	Institute of Environmental Protection-Puszcza Borecka station	Kolektorska 4, 01-692, Warszawa, Poland
8	Ireland	EPA, Dublin Inspectorate McCumiskey Hs,	Richview, Clonskeagh Rd, Dublin, D14YR62, Ireland
9	United Kingdom	Natural Resources Wales Analytical Seervices (NRWAS)	As per delivery address below
10	Ireland	Environment Protection Agency	The Glen Monaghan H18 YT02
11	Ireland	EPA Regional Inspectorate Castlebar OEA	John Moore Road, Castlebar, Ireland.
12	Russian Federation	Institute of Global Climate and Ecology (IGCE) Roshydromet and RAS Russian Academy of Sciences	20-B, Glebovskaya St., Moscow, 107258
13	Estonia	Estonian Environment Research Centre	Marja 4 D 10617 Tallinn Estonia
14	Netherlands	Radbouduniversiteit afd. Ecologie t.a.v. G. Verheggen	Postbus 9010 6500 GL Nijmegen The Netherlands
15	Poland	Polish Academy of Sciences Institute of Botany	PAN Instytut Botaniki 31-512 Kraków ul. Lubicz 46
16	Lithuania	Environmental Protection Agency Research Department	A.Gostauto 9 01108 Vilnius
17	Germany	Staatliche Betriebgesellschaft für Umwelt und Landwirtschaft (BfUL)	Stephanplatz 3 D-09112 Chemnitz
18	Belgium	Vlaamse MilieuMaatschappij (VMM) Dienst Laboratorium	Raymonde de Larochelaan 1,9051 Sint-Denijs-Westrem

No	Laboratory	Town	Country
19	Czech Republic	Chemical Laboratory, Czech Geological Survey	Geologická 6, 152 00 Prague
20	Russian Federation	Institute of Industrial Ecology Problems of the North (INEP) Group ICP methods of analysis	184209 Apatity, Akademgorodok 14A, Murmansk reg.
21	Russian Federation	FGU «Baltwodhoz»	Saint-Petersburg, V.O. Sredny pr. 26
22	Sweden	Swedish University for Agricultural Sciences Aquatic Sciences and Assessment	Box 7050 750 07 UPPSALA
23	Germany	Forest Nutrition and Water Resources Department of Ecology, Technis	H.C.v. Carlowitz-Platz 2 D-85354 Freising Germany
24	Finland	University of Helsinki Lab. of Geology and Geography	P.O. Box 64 00014 University of Helsinki
25	Germany	Staatliche Betriebgesellschaft für Umwelt und Landwirtschaft (BfUL)	Haus5, FB53 Waldheimer Str. 219 D-01683 Nossen
26	Moldova	State Hydrometeorological Service EQMD	134 Grenoble Str, Chisinau Moldova, Republic MD-2072
27	United Kingdom	Marine Scotland Science Freshwater Laboratory	Faskally, Pitlochry, Perthshire,PH16 5BB, Scotland.
28	Serbia	Institute for Public Health Pancevo	Pasterova 2 26000 Pancevo
29	Spain	Servei d'Anàlisi Química i Estructural	STR-UdG Pic de Peguera, 15 17003-Girona
30	Canada	MOEECC, DORSET Laboratory	P.O. Box 39 Dorset, Ontario Canada P0A 1E0
31	Austria	Institut fur Ökologie	Technikerstrasse 25 6020 Innsbruck Austria
32	Belgium	ISSeP Colfontaine Zoning Schweitzer	Rue de la Platinerie B-7340 COLFONTAINE
33	Switzerland	Ufficio del Monitoraggio Ambientale - Laboratorio	Via Mirasole 22 6500 Bellinzona
34	France	Laboratoire d'Ecologie Fonctionnelle et Environnement (ECOLAB)	Avenue Agrobiopole 31326 Castanet Tolosan
35	Italy	Lab di Microanalysis DISPAA University of Florence	Via della Lastruccia,13 50019SestoF.no Firenze
36	Ireland	Kilkenny Lab, Environmental Protection agency	Environmental Protection Agency, Seville Lodge,

No	Laboratory	Town	Country
37	Russian Federation	Hydrochemical Laboratory by Federal State Enterprise on Water Industry	10 A Stahanovskaya str., Pskov, 180004
38	United Kingdom	NLS Starcross laboratory Staplake Mount	Starcross, Exeter, Devon, EX6 8FD

Number of participating laboratories from the different countries represented in intercomparison 1731

Country	No. of labs.	Country	No. of labs.
Austria	1	Netherlands	1
Belgium	2	Norway	1
Canada	1	Poland	2
Czech Republic	1	Moldova	1
Estonia	1	Russia	5
Finland	1	Serbia	1
France	1	Spain	1
Germany	6	Sweden	1
Ireland	4	Switzerland	1
Italy	2	United Kingdom	3
Lithuania	1		

Total: 21 countries

Appendix B.

Preparation of samples

The sample solutions were prepared from water collected from Sogsnvann lake, located in the Oslo municipality (right in the North greenbelt around Oslo city). This lake is a popular recreational area for the residents of Oslo during the summer, as well as a cross-country skiing, skating and ice fishing destination in the winter. The water, collected in 25 litre plastic containers, was brought to the laboratory and stored for about two weeks. The water was then filtrated through 0.45 μm cellulose acetate membrane. The filtrate was collected in polyethylene containers and stored at room temperature one more week to equilibrate. Small aliquots were taken from the filtrate to determine the background concentrations of the analytical variables of interest.

In the current edition, sample set AB was obtained lowering the natural pH of the effluent by addition of HCl and H_2SO_4 diluted solutions. The TOC content was slightly increased by adding a few drops of a concentrated solution of humic acid. Phosphorous was added in organic form using a standard solution of inositol hexaphosphate (phytic acid).

The samples for the set CD were prepared by spiking the filtered water with stock solutions of stoichiometric compounds containing heavy metals and preserved by addition of 5 ml concentrated nitric acid pr. liter sample to yield a 0.5% v/v concentration.

A few days before shipping the samples to the participants, they were transferred to 500 ml (sample set AB) or 250 ml acid washed (sample set CD) high density polyethylene bottles with screw cap. These samples were stored at room temperature until they were delivered to the participating laboratories.

Appendix C.

Treatment of analytical data

The intercomparison was carried out by the method of Youden. This procedure requires two samples to be analyzed, and each laboratory shall report only one result per sample and analytical variable. In a coordinate system, the result of sample B is plotted against the result of sample A (see Figures 1 - 20).

The Youden's chart allows the possibility to distinguish between random and systematic errors affecting the results. The two straight lines drawn in the diagram represent the true values of the samples; or - as in this case, when the true value is not known - the median value of the results from the participating laboratories. The results being omitted in the statistical calculations are not used in the determination of the median value and thus, the true value. The diagram is thus divided into four quadrants. In a hypothetical case, when the analysis is affected by random errors only, the results will spread randomly over the four quadrants.

However, the results are usually located in the lower left and the upper right quadrant, constituting a characteristic elliptical pattern along the 45° line. This is reflecting the fact that many laboratories - due to systematic deviations - have attained too low or too high values for both samples.

The acceptance limit of the results may be represented by a circle with its centre at the intersection of the two straight lines in the diagram (true or median values). The distance between the centre of the circle and the mark representing the laboratory is a measure of the total error of the results. The distance along the 45° line gives the magnitude of the systematic error, while the distance perpendicular to the 45° line indicates the magnitude of the random error. The location of the laboratory in the Youden's diagram provides then important information about the size and type of analytical error, making it easier to ascertain which the source of error is.

The statistical treatment of the analytical results was accomplished in this way: Pairs of results where one or both of the values lie outside the true value $\pm$ 50 % are omitted from the statistical calculations. The remaining results are used for the calculation of the mean value (x) and the standard deviation (s). Now the pairs of results where one or both of the values are lying outside x $\pm$ 3s, are omitted. The remaining results are used for a final calculation, the results of which are presented in the tables 5.1 - 5.20. Results being omitted from the calculations are marked with the letter "O".

Estimation of uncertainty of the true values

The median value of the reported results, after exclusion of strongly deviating results, is used as the true value for this intercomparison. Thus, the true value is based upon consensus value from the participants and therefore, the estimation of the uncertainty of the true value could be based on the method given in ISO 13528 (2005), Annex C (algorithm A).

For each parameter the median value is determined and an initial value for the robust standard deviation is calculated from the absolute differences between the median value and the result of each participating laboratory according to:

$S^* = 1{,}483 \times$ the median of $|x_i - m|$ $\quad (i = 1, 2 \ldots p)$

New value for the robust standard deviation is then calculated according to equations C.3-C6 in Annex C. The robust standard deviation is then derived by an iterative calculation by updating the values several times using the modified data, until the process converges.

The uncertainty u_X of the assigned value for the true value is then calculated according to chapter 5.6 in ISO 13528:

$$u_X = 1{,}25 x S^* / \sqrt{p}$$

For the estimation of expanded uncertainty U, a coverage factor of two is used:

$U = 2 \times u_X$

It is important to know that there are some limitations in this approach for the estimation of the uncertainty of the true value:

- There may be no real consensus among the participants
- The consensus may be biased by the general use of faulty methodology and this bias will not be reflected in the standard uncertainty of the assigned value using this calculation.

Table 3. Estimation of uncertainty of the assigned true values

Parameter and unit	Sample	True value	Total no.	Robust std.dev.	Uncertainty	Expanded uncertainty
pH	A	5.78	31	0.252	0.056	0.113
	B	5.76	31	0.228	0.051	0.102
Conductivity	A	2.90	27	0.087	0.021	0.042
(mS/m)	B	2.55	26	0.054	0.013	0.027
Alkalinity	A	0.022	11	0.0060	0.0023	0.0045
(mmol/l)	B	0.020	9	0.0020	0.0008	0.0017
Nitrate + nitrite-nitrogen	A	66	14	11.3	3.8	7.6
(µg N/l)	B	61	13	10.7	3.7	7.4
Chloride	A	2.0	22	0.12	0.03	0.07
(mg/l)	B	1.8	22	0.11	0.03	0.06
Sulphate	A	5.62	27	0.186	0.045	0.089
(mg/l)	B	5.00	27	0.210	0.050	0.101
Calcium	A	2.40	30	0.199	0.045	0.091
(mg/l)	B	2.09	28	0.139	0.033	0.066
Magnesium	A	0.37	29	0.017	0.004	0.008
(mg/l)	B	0.32	28	0.012	0.003	0.006
Sodium	A	1.63	28	0.100	0.024	0.047
(mg/l)	B	1.44	26	0.081	0.020	0.040
Potassium	A	0.23	26	0.027	0.007	0.013
(mg/l)	B	0.21	26	0.020	0.005	0.010
Total organic carbon	A	15.73	19	1.555	0.446	0.892
(mg/l)	B	14.30	19	1.423	0.408	0.816
Total P	A	11.50	14	3.974	1.328	2.655
(µg/l)	B	10.00	9	1.844	0.768	1.536
Aluminium	C	178	21	13.2	3.6	7.2
(µg/l)	D	157	21	11.6	3.2	6.3
Iron	C	81.75	22	3.550	0.946	1.892
(µg/l)	D	73.05	22	4.522	1.205	2.410
Manganese	C	48.90	23	1.688	0.440	0.880
(µg/l)	D	43.00	23	1.686	0.439	0.879
Cadmium	C	9.68	23	0.603	0.157	0.314
(µg/l)	D	8.59	23	0.535	0.139	0.279
Lead	C	7.82	22	0.619	0.165	0.330
(µg/l)	D	6.82	22	0.399	0.106	0.213
Copper	C	29.63	22	1.251	0.333	0.667
(µg/l)	D	26.95	22	1.122	0.299	0.598
Nickel	C	14.29	23	0.720	0.188	0.376
(µg/l)	D	12.74	23	0.626	0.163	0.327
Zinc	C	19.68	22	0.757	0.202	0.403
(µg/l)	D	18.42	22	0.665	0.177	0.354

Appendix D

Table 4. The results of the participating laboratories.

Lab. nr.	pH		Conductivity, mS/m		Alkalinity, mmol/l		Nitrate + nitrite-nitrogen, µg N/l		Chloride, mg/l		Sulphate, mg/l	
	A	B	A	B	A	B	A	B	A	B	A	B
1	5.95	6.09	2.86	2.52	0.070	0.064	2	32	1.9	1.6	5.52	4.84
2	5.78	5.86	2.86	2.52			0	0			5.63	4.82
3	5.79	5.75	2.84	2.52	0.022	0.019	75	67	2.0	1.8	5.58	4.98
4	5.85	5.92	2.90	2.54			66	61	2.0	1.8	5.58	5.16
5	5.51	5.63	2.93	2.62	0.057	0.057			2.2	1.9	6.01	5.23
6	6.13	6.16	2.69	2.39			66	59	1940.0	1719.0	5.52	4.87
7	6.12	6.18	2.89	2.56								
8	5.84	5.87	2.90	2.55					2070.0	1730.0	5.48	4.80
9	6.08	6.02	2.56	2.28	0.018	0.017	180	200	1220.0	920.0	5.86	5.15
10	5.98	5.95	3.10	2.60	0.054	0.052	140	90	2.6	2.3		
11	5.80	5.80	49.80	30.10	2.800	2.700	60	50	1.9	1.7		
12												
13	5.77	5.75	2.87	2.53	0.019	0.016	66	68	2.0	1.9	5.85	5.20
14	5.15	5.24			0.367	0.038	65	61	1.8	1.4		
15	5.67	5.69	2.89	2.56			61	55	2.2	1.6	5.62	5.08
16	7.54	6.94	2.94	2.53								
17	5.90	5.90	3.00	2.60	0.037	0.035	80	70	1.7	1.5	5.40	4.90
18												
19	5.96	5.96	3.05	2.91	0.029	0.026			1.9	1.7	5.63	4.90
20	5.64	5.69	2.77	2.54	0.015	0.016	81	72	2.0	1.8	5.72	5.00
21	5.68	5.58	2.93	2.58	0.000	0.000	52	42	2.1	1.8	5.57	4.76
22	5.52	5.61	2.79	2.47	0.018	0.016	95	84	2.0	1.8	5.70	5.00
23	5.45	5.38	2.93	2.61	0.215	0.103	230	200	2.0	1.7	5.43	4.62
24	5.72	5.88	29.20	25.70	0.010	0.015	52	31	1.9	1.7	5.51	4.66
25	5.70	5.73	2.91	2.59			100	90	2.0	1.8	5.70	5.12
26	5.26	5.31	0.29	0.26	0.030	0.020	100	130	7800.0	7090.0	3.60	2.70
27	5.64	5.67	2.75	2.41	0.020	0.020	0	0	2.0	1.8	5.77	5.06
28											10.56	4.31
29												
30	6.73	5.94	2.90	2.56	0.046	0.023	100	87	1942.0	1715.0	5.51	4.91
31	5.78	5.76	2.90	2.56	0.020	0.017	71	62	2.0	1.8	5.78	5.06
32	5.71	5.70	2.91	2.57								
33	5.80	5.83	2.85	2.53	0.019	0.018	886	722	2.3	2.0	5.96	5.31
34	5.82	5.84	3.26	2.80	0.030	0.250	67	56	1.9	1.7	5.54	5.07
35												
36	5.40	5.50	30.00	26.00	3.278	2.350	0	0	1.6	1.8	4.54	4.40
37	5.42	5.45	2.85	2.50	0.043	0.041	0	0	2.0	1.8	5.50	5.00
38											6.49	5.54

Lab. nr.	Sodium mg/l		Potassium mg/l		TOC mg/l		Total P µg/l		Aluminium µg/l		Iron µg/l	
	A	B	A	B	A	B	A	B	A	B	A	B
1	1.68	1.49	0.16	0.14	13.20	15.50	7.00	10.00	180	157	80.30	73.10
2	1.52	1.40	0.25	0.22	15.59	13.58	22.23	20.44	218	196	81.80	71.60
3	1.81	1.60	0.23	0.21	15.00	12.90	10.00	7.00	160	143	82.00	73.00
4	1.50	1.30	0.22	0.19	16.00	16.30			171	151	83.90	74.40
5	1.51	1.34	0.24	0.21	10.40	9.78	8.17	7.39	172	156	84.53	75.42
6	1.72	1.52	0.22	0.22	14.98	12.33	-20.00	-20.00	165	149	78.40	69.70
7												
8	2.18	1.75	0.26	0.21					171	156	77.64	61.10
9	1.55	1.37	0.23	0.20	15.45	13.62	17.20	20.50	186	164	71.00	55.08
10					17.00	14.29	16.00	12.00				
11					17.00	15.00	17.00	10.00				
12												
13	1.61	1.42	0.23	0.21	13.60	12.40			184	163	86.70	101.00
14	0.96	0.74	0.22	0.20								
15	1.56	1.38	0.28	0.25					185	175	79.80	72.20
16												
17	2.00	1.80	0.00	0.00	3.90	4.40						
18	2.43	2.13	0.26	0.23	16.32	14.37			180	157		
19	1.49	1.44	0.28	0.22			11.70	10.90	124	115	110.00	80.00
20	1.72	1.53	0.28	0.24			14.00	12.00	163	149	79.10	70.10
21	1.60	1.40	0.24	0.21	15.40	14.30	11.30	10.40	189	168	80.20	71.90
22	1.70	1.50	0.23	0.21	17.40	15.30	10.30	9.30	170	150	83.00	74.00
23	1.57	1.36	0.20	0.17	7.51	6.32	15.00	10.00				
24	1.64	1.44	0.24	0.21			16.00	13.00	192	165	87.00	78.00
25	1.66	1.46	0.23	0.20								
26	1.70	1.40	0.50	0.25			4.00	5.00			100.00	85.00
27	1.67	1.46	0.28	0.24	16.88	15.11	8.03	8.45				
28									382	362		
29	1.68	1.47	0.25	0.22					189	164	63.82	61.24
30	1.63	1.45	0.36	0.22	16.40	14.50						
31	1.62	1.43	0.25	0.22	15.87	14.15	10.90	9.20				
32											81.94	72.70
33	1.62	1.58	0.22	0.18	16.79	15.13			178	161	81.58	74.31
34	1.57	1.41	0.20	0.18	16.45	14.63						
35									151	132	80.66	70.87
36					11.00	12.00	0.01	0.01				
37	1.54	1.28	0.23	0.22			17.90	13.20	182	169	82.20	74.70
38	1.61	1.44	0.23	0.20					178	158	81.70	74.50

Lab. nr.	Manganese µg/l		Cadmium µg/l		Lead µg/l		Copper µg/l		Nickel µg/l		Zinc µg/l	
	A	B	A	B	A	B	A	B	C	D	C	D
1	48.20	42.60	9.60	8.64	8.13	8.90	29.10	27.00	14.00	12.40	19.70	18.30
2	51.03	45.20	9.90	8.82	7.84	6.58	31.35	28.29	14.52	12.67	20.38	19.03
3	49.00	43.00	8.90	7.80	6.90	6.20	30.00	27.00	13.20	12.00	18.50	18.00
4	48.80	43.30	10.40	9.19	8.97	7.49	31.40	27.90	14.70	13.00	21.30	20.30
5	51.04	44.63	9.81	8.73	7.58	6.47	28.54	25.81	15.18	13.63	19.68	18.64
6	46.30	40.60	9.55	8.56	7.82	6.69	29.13	26.35	14.01	12.40	19.05	18.42
7												
8	47.21	41.69	9.13	8.15	8.06	7.04	29.00	25.82	13.90	12.13	19.17	18.42
9	49.70	42.61	9.70	8.58	7.72	6.66			15.40	13.30	20.50	19.30
10												
11												
12			10.70	8.90	4.77	3.74						
13	48.90	43.80	9.76	8.66	8.00	7.02	30.40	27.40	14.80	13.10	19.30	18.40
14												
15	48.10	43.20	9.45	8.13	7.45	6.80	28.70	26.00	12.30	11.60	20.20	17.10
16												
17												
18	47.89	42.05	9.62	8.46	8.56	7.41	29.57	26.70	13.59	12.07	17.88	16.29
19	48.00	43.00	9.07	8.06	8.70	7.10	33.20	29.50	14.50	13.30	45.00	22.70
20	46.90	41.50	8.81	7.79	7.63	6.65	30.60	27.30	13.60	12.00	21.80	20.10
21	49.40	43.60	9.80	8.72	7.63	7.15	29.50	26.20	14.70	12.90	19.30	18.20
22	49.00	44.00	9.80	8.60	8.20	7.20	30.00	27.00	14.00	13.00	20.00	19.00
23												
24	49.00	44.00	10.00	9.10	9.10	7.10	31.00	28.00	15.00	13.00	19.00	19.00
25												
26	49.93	41.11	5.95	5.69	2.87	2.26	28.81	24.53	13.97	12.67		
27												
28												
29	49.37	43.30	7.46	6.63	7.88	6.89	29.06	25.78	14.29	12.74	19.88	18.68
30												
31												
32	47.06	40.93	8.94	7.52	7.42	6.31	28.63	26.05	13.00	10.99	19.25	18.37
33			10.43	9.15	9.16	6.82	32.12	28.05	14.43	13.19	19.67	18.98
34												
35	45.02	39.40	8.71	7.93	7.16	6.48	25.44	22.78	13.53	11.88	20.57	16.96
36												
37	54.10	45.80	9.70	8.39	7.51	6.94	29.70	27.10	14.60	12.90	19.20	18.30
38	51.10	46.80	9.66	8.66	7.78	6.67	30.00	26.90	14.40	12.80	20.10	18.90

Table 5.1. Statistics. pH
Sample A

Analytical method: All
Unit: units

Number of participants	32	Range	0.98
Number of omitted results	2	Variance	0.06
True value	5.78	Standard deviation	0.24
Mean value	5.73	Relative standard deviation	4.1%
Median value	5.78	Relative error	-0.9%

Analytical results in ascending order:

14	5.15	25	5.70	4	5.85	
26	5.26	32	5.71	17	5.90	
36	5.40	24	5.72	1	5.95	
37	5.42	13	5.77	19	5.96	
23	5.45	2	5.78	10	5.98	
5	5.51	31	5.78	9	6.08	
22	5.52	3	5.79	7	6.12	
27	5.64	11	5.80	6	6.13	
20	5.64	33	5.80	30	6.73	O
15	5.67	34	5.82	16	7.54	O
21	5.68	8	5.84			

O = Omitted result

Table 5.1. Statistics. pH
Sample B

Analytical method: All
Unit: units

Number of participants	32	Range	0.94
Number of omitted results	2	Variance	0.05
True value	5.76	Standard deviation	0.23
Mean value	5.76	Relative standard deviation	4.0%
Median value	5.76	Relative error	-0.1%

Analytical results in ascending order:

14	5.24	32	5.70	17	5.90	
26	5.31	25	5.73	4	5.92	
23	5.38	13	5.75	30	5.94	O
37	5.45	3	5.75	10	5.95	
36	5.50	31	5.76	19	5.96	
21	5.58	11	5.80	9	6.02	
22	5.61	33	5.83	1	6.09	
5	5.63	34	5.84	6	6.16	
27	5.67	2	5.86	7	6.18	
15	5.69	8	5.87	16	6.94	O
20	5.69	24	5.88			

O = Omitted result

Table 5.2. Statistics. Conductivity
Sample A

Analytical method: All
Unit: mS/m

Number of participants	31	Range	2.97
Number of omitted results	3	Variance	0.26
True value	2.90	Standard deviation	0.51
Mean value	2.80	Relative standard deviation	18.1%
Median value	2.90	Relative error	-3.5%

Analytical results in ascending order:

26	0.29	13	2.87	23	2.93	
9	2.56	15	2.89	16	2.94	
6	2.69	7	2.89	17	3.00	
27	2.75	4	2.90	19	3.05	
20	2.77	30	2.90	10	3.10	
22	2.79	31	2.90	34	3.26	
3	2.84	8	2.90	24	29.20	O
37	2.85	25	2.91	36	30.00	O
33	2.85	32	2.91	11	49.80	O
1	2.86	21	2.93			
2	2.86	5	2.93			

O = Omitted result

Table 5.2. Statistics. Conductivity
Sample B

Analytical method: All
Unit: mS/m

Number of participants	31	Range	2.65
Number of omitted results	3	Variance	0.20
True value	2.55	Standard deviation	0.45
Mean value	2.47	Relative standard deviation	18.1%
Median value	2.55	Relative error	-3.1%

Analytical results in ascending order:

26	0.26	33	2.53	17	2.60	
9	2.28	4	2.54	10	2.60	
6	2.39	20	2.54	23	2.61	
27	2.41	8	2.55	5	2.62	
22	2.47	31	2.56	34	2.80	
37	2.50	30	2.56	19	2.91	
1	2.52	7	2.56	24	25.70	O
2	2.52	15	2.56	36	26.00	O
3	2.52	32	2.57	11	30.10	O
13	2.53	21	2.58			
16	2.53	25	2.59			

O = Omitted result

Table 5.3. Statistics. Alkalinity

Sample A

Analytical method: All

Unit: mmol/L

Number of participants	23	Range	0.070
Number of omitted results	5	Variance	0.000
True value	0.022	Standard deviation	0.018
Mean value	0.029	Relative standard deviation	62.7%
Median value	0.021	Relative error	33.1%

Analytical results in ascending order:

21	0.000		27	0.020		10	0.054	
24	0.010		3	0.022		5	0.057	
20	0.015		19	0.029		1	0.070	
9	0.018		34	0.030	O	23	0.215	O
22	0.018		26	0.030		14	0.367	O
13	0.019		17	0.037		11	2.800	O
33	0.019		37	0.043		36	3.278	O
31	0.020		30	0.046				

O = Omitted result

Table 5.3. Statistics. Alkalinity

Sample B

Analytical method: All

Unit: mmol/L

Number of participants	23	Range	0.064
Number of omitted results	5	Variance	0.000
True value	0.020	Standard deviation	0.017
Mean value	0.026	Relative standard deviation	64.3%
Median value	0.019	Relative error	32.3%

Analytical results in ascending order:

21	0.000		3	0.019		10	0.052	
24	0.015		27	0.020		5	0.057	
22	0.016		26	0.020		1	0.064	
13	0.016		30	0.023		23	0.103	O
20	0.016		19	0.026		34	0.250	O
9	0.017		17	0.035		36	2.350	O
31	0.017		14	0.038	O	11	2.700	O
33	0.018		37	0.041				

O = Omitted result

Table 5.4. Statistics. Nitrate + nitrite-nitrogen

Sample A

Analytical method: All
Unit: microg/L

Number of participants	26	Range	230
Number of omitted results	1	Variance	2967
True value	66	Standard deviation	54
Mean value	72	Relative standard deviation	75.3%
Median value	66	Relative error	9.6%

Analytical results in ascending order:

36	0	14	65	22	95	
2	0	13	66	25	100	
37	0	4	66	26	100	
27	0	6	66	30	100	
1	2	34	67	10	140	
24	52	31	71	9	180	
21	52	3	75	23	230	
11	60	17	80	33	886	O
15	61	20	81			

O = Omitted result

Table 5.4. Statistics. Nitrate + nitrite-nitrogen

Sample B

Analytical method: All
Unit: microg/L

Number of participants	26	Range	200
Number of omitted results	1	Variance	2623
True value	61	Standard deviation	51
Mean value	67	Relative standard deviation	76.8%
Median value	61	Relative error	9.3%

Analytical results in ascending order:

36	0	34	56	22	84	
2	0	6	59	30	87	
37	0	14	61	25	90	
27	0	4	61	10	90	
24	31	31	62	26	130	
1	32	3	67	9	200	
21	42	13	68	23	200	
11	50	17	70	33	722	O
15	55	20	72			

O = Omitted result

Table 5.5. Statistics. Chloride

Sample A

Analytical method: All
Unit: mg/L

Number of participants	28	Range	2068.4
Number of omitted results	1	Variance	437647.3
True value	2.0	Standard deviation	661.5
Mean value	267.3	Relative standard deviation	247.5%
Median value	2.0	Relative error	13266.4%

Analytical results in ascending order:

36	1.6	23	2.0	5	2.2	
17	1.7	27	2.0	33	2.3	
14	1.8	31	2.0	10	2.6	
1	1.9	37	2.0	9	1220.0	
11	1.9	22	2.0	6	1940.0	
24	1.9	25	2.0	30	1942.0	
34	1.9	13	2.0	8	2070.0	
19	1.9	4	2.0	26	7800.0	O
20	2.0	21	2.1			
3	2.0	15	2.2			

O = Omitted result

Table 5.5. Statistics. Chloride

Sample B

Analytical method: All
Unit: mg/L

Number of participants	28	Range	1728.6
Number of omitted results	1	Variance	321014.6
True value	1.8	Standard deviation	566.6
Mean value	226.8	Relative standard deviation	249.8%
Median value	1.8	Relative error	12715.2%

Analytical results in ascending order:

14	1.4	3	1.8	5	1.9	
17	1.5	21	1.8	33	2.0	
15	1.6	20	1.8	10	2.3	
1	1.6	4	1.8	9	920.0	
19	1.7	31	1.8	30	1715.0	
24	1.7	36	1.8	6	1719.0	
34	1.7	22	1.8	8	1730.0	
23	1.7	37	1.8	26	7090.0	O
11	1.7	25	1.8			
27	1.8	13	1.9			

O = Omitted result

Table 5.6. Statistics. Sulphate

Sample A

Analytical method: All

Unit: mg/L

Number of participants	29	Range	1.09
Number of omitted results	3	Variance	0.05
True value	5.62	Standard deviation	0.23
Mean value	5.68	Relative standard deviation	4.0%
Median value	5.63	Relative error	1.0%

Analytical results in ascending order:

26	3.60	O	34	5.54	9	5.75	
36	4.54	O	21	5.57	27	5.77	
17	5.40		3	5.58	31	5.78	
23	5.43		4	5.58	13	5.85	
8	5.48		15	5.62	9	5.86	
37	5.50		19	5.63	33	5.96	
30	5.51		2	5.63	5	6.01	
24	5.51		22	5.70	38	6.49	
1	5.52		25	5.70	28	10.56	O
6	5.52		20	5.72			

O = Omitted result

Table 5.6. Statistics. Sulphate

Sample B

Analytical method: All

Unit: mg/L

Number of participants	29	Range	0.92
Number of omitted results	3	Variance	0.04
True value	5.00	Standard deviation	0.20
Mean value	5.01	Relative standard deviation	4.1%
Median value	5.00	Relative error	0.1%

Analytical results in ascending order:

26	2.70	O	19	4.90	15	5.08
28	4.31	O	17	4.90	25	5.12
36	4.40	O	30	4.91	9	5.13
23	4.62		3	4.98	9	5.15
24	4.66		22	5.00	4	5.16
21	4.76		37	5.00	13	5.20
8	4.80		20	5.00	5	5.23
2	4.82		31	5.06	33	5.31
1	4.84		27	5.06	38	5.54
6	4.87		34	5.07		

O = Omitted result

Table 5.7. Statistics. Calcium

Sample A

Analytical method: All
Unit: mg/L

Number of participants	30	Range	1.51
Number of omitted results	0	Variance	0.10
True value	2.40	Standard deviation	0.32
Mean value	2.42	Relative standard deviation	13.4%
Median value	2.40	Relative error	1.0%

Analytical results in ascending order:

34	1.70	33	2.34	30	2.47
37	1.83	15	2.35	8	2.48
38	2.04	32	2.37	22	2.50
29	2.16	19	2.37	13	2.54
9	2.23	5	2.40	31	2.55
21	2.24	20	2.40	25	2.57
4	2.27	27	2.43	18	2.77
1	2.28	24	2.43	14	3.04
3	2.30	23	2.46	17	3.20
2	2.32	6	2.46	26	3.21

O = Omitted result

Table 5.7. Statistics. Calcium

Sample B

Analytical method: All
Unit: mg/L

Number of participants	30	Range	1.14
Number of omitted results	0	Variance	0.06
True value	2.09	Standard deviation	0.24
Mean value	2.12	Relative standard deviation	11.3%
Median value	2.09	Relative error	1.6%

Analytical results in ascending order:

37	1.66	3	2.05	5	2.17
34	1.67	2	2.06	23	2.17
29	1.89	19	2.07	18	2.19
38	1.90	32	2.07	22	2.20
1	1.95	30	2.08	25	2.25
21	1.97	20	2.10	31	2.27
4	1.98	27	2.11	13	2.29
8	2.03	9	2.14	14	2.54
15	2.05	24	2.14	17	2.70
33	2.05	6	2.16	26	2.80

O = Omitted result

Table 5.8. Statistics. Magnesium

Sample A

Analytical method: All

Unit: mg/L

Number of participants	30	Range	0.07
Number of omitted results	2	Variance	0.00
True value	0.37	Standard deviation	0.02
Mean value	0.37	Relative standard deviation	4.5%
Median value	0.37	Relative error	0.1%

Analytical results in ascending order:

9	0.35	2	0.36	3	0.37	
38	0.35	23	0.37	31	0.38	
1	0.35	33	0.37	37	0.38	
21	0.35	30	0.37	18	0.39	
6	0.35	13	0.37	32	0.39	
15	0.36	4	0.37	8	0.40	
5	0.36	27	0.37	14	0.41	
22	0.36	24	0.37	17	0.41	
19	0.36	25	0.37	34	0.41	O
29	0.36	20	0.37	26	0.97	O

O = Omitted result

Table 5.8. Statistics. Magnesium

Sample B

Analytical method: All

Unit: mg/L

Number of participants	30	Range	0.05
Number of omitted results	2	Variance	0.00
True value	0.32	Standard deviation	0.01
Mean value	0.32	Relative standard deviation	3.8%
Median value	0.32	Relative error	0.7%

Analytical results in ascending order:

1	0.30	19	0.32	3	0.33	
17	0.30	25	0.32	37	0.33	
29	0.31	4	0.32	13	0.33	
21	0.31	22	0.32	31	0.34	
6	0.31	27	0.32	18	0.34	
15	0.31	30	0.32	14	0.34	
38	0.31	2	0.32	32	0.35	
33	0.32	23	0.33	20	0.35	
9	0.32	5	0.33	34	0.38	O
8	0.32	24	0.33	26	0.97	O

O = Omitted result

Table 5.9. Statistics. Sodium

Sample A

Analytical method: All

Unit: mg/L

Number of participants	29	Range	0.69
Number of omitted results	2	Variance	0.02
True value	1.63	Standard deviation	0.15
Mean value	1.65	Relative standard deviation	9.0%
Median value	1.62	Relative error	1.8%

Analytical results in ascending order:

14	0.96	O	21	1.60	1	1.68		
19	1.49		13	1.61	22	1.70		
4	1.50		38	1.61	26	1.70		
5	1.51		31	1.62	20	1.72		
2	1.52		33	1.62	6	1.72		
37	1.54		30	1.63	3	1.81		
9	1.55		24	1.64	17	2.00		
15	1.56		25	1.66	8	2.18		
23	1.57		27	1.67	18	2.43	O	
34	1.57		29	1.68				

O = Omitted result

Table 5.9. Statistics. Sodium

Sample B

Analytical method: All

Unit: mg/L

Number of participants	29	Range	0.52
Number of omitted results	2	Variance	0.01
True value	1.44	Standard deviation	0.12
Mean value	1.46	Relative standard deviation	8.0%
Median value	1.44	Relative error	1.4%

Analytical results in ascending order:

14	0.74	O	34	1.41	1	1.49	
37	1.28		13	1.42	22	1.50	
4	1.30		31	1.43	6	1.52	
5	1.34		19	1.44	20	1.53	
23	1.36		24	1.44	33	1.58	
9	1.37		38	1.44	3	1.60	
15	1.38		30	1.45	8	1.75	
21	1.40		25	1.46	17	1.80	
26	1.40		27	1.46	18	2.13	O
2	1.40		29	1.47			

O = Omitted result

Table 5.10. Statistics. Potassium

Sample A

Analytical method: All
Unit: mg/L

Number of participants	29	Range	0.08
Number of omitted results	4	Variance	0.00
True value	0.23	Standard deviation	0.02
Mean value	0.24	Relative standard deviation	9.6%
Median value	0.23	Relative error	3.0%

Analytical results in ascending order:

17	0.00	O	3	0.23		2	0.25	
1	0.16	O	25	0.23		8	0.26	
23	0.20		38	0.23		18	0.26	
34	0.20		37	0.23		15	0.28	
33	0.22		13	0.23		20	0.28	
14	0.22		5	0.24		27	0.28	
4	0.22		21	0.24		19	0.28	
6	0.22		24	0.24		30	0.36	O
9	0.23		31	0.25		26	0.50	O
22	0.23		29	0.25				

O = Omitted result

Table 5.10. Statistics. Potassium

Sample B

Analytical method: All
Unit: mg/L

Number of participants	29	Range	0.08
Number of omitted results	4	Variance	0.00
True value	0.21	Standard deviation	0.02
Mean value	0.21	Relative standard deviation	8.9%
Median value	0.21	Relative error	0.2%

Analytical results in ascending order:

17	0.00	O	13	0.21		6	0.22	
1	0.14	O	24	0.21		19	0.22	
23	0.17		3	0.21		37	0.22	
33	0.18		8	0.21		29	0.22	
34	0.18		21	0.21		18	0.23	
4	0.19		22	0.21		20	0.24	
14	0.20		5	0.21		27	0.24	
9	0.20		30	0.22	O	15	0.25	
25	0.20		2	0.22		26	0.25	O
38	0.20		31	0.22				

O = Omitted result

Table 5.11. Statistics. Total organic carbon

Sample A

Analytical method: All

Unit: mg/L

Number of participants	21	Range	7.00
Number of omitted results	2	Variance	3.86
True value	15.73	Standard deviation	1.96
Mean value	15.30	Relative standard deviation	12.8%
Median value	15.87	Relative error	-2.7%

Analytical results in ascending order:

17	3.90	O	3	15.00		30	16.40
23	7.51	O	21	15.40		34	16.45
5	10.40		9	15.45		33	16.79
36	11.00		2	15.59		27	16.88
1	13.20		31	15.87		11	17.00
13	13.60		4	16.00		10	17.00
6	14.98		18	16.32		22	17.40

O = Omitted result

Table 5.11. Statistics. Total organic carbon

Sample B

Analytical method: All

Unit: mg/L

Number of participants	21	Range	6.52
Number of omitted results	2	Variance	2.35
True value	14.30	Standard deviation	1.53
Mean value	13.96	Relative standard deviation	11.0%
Median value	14.30	Relative error	-2.4%

Analytical results in ascending order:

17	4.40	O	2	13.58		34	14.63
23	6.32	O	9	13.62		11	15.00
5	9.78		31	14.15		27	15.11
36	12.00		10	14.29		33	15.13
6	12.33		21	14.30		22	15.30
13	12.40		18	14.37		1	15.50
3	12.90		30	14.50		4	16.30

O = Omitted result

Table 5.12. Statistics. Total P

Sample A

Unit: microg/L

Number of participants	19	Range	22.22
Number of omitted results	1	Variance	29.90
True value	11.50	Standard deviation	5.47
Mean value	12.04	Relative standard deviation	45.4%
Median value	11.50	Relative error	4.7%

Analytical results in ascending order:

6	-20.00	O	22	10.30	10	16.00
36	0.01		31	10.90	11	17.00
26	4.00		21	11.30	9	17.20
1	7.00		19	11.70	37	17.90
27	8.03		20	14.00	2	22.23
5	8.17		23	15.00		
3	10.00		24	16.00		

O = Omitted result

Table 5.12. Statistics. Total P

Sample D

Analytical method: All

Unit: microg/L

Number of participants	19	Range	20.49
Number of omitted results	1	Variance	22.78
True value	10.00	Standard deviation	4.77
Mean value	10.49	Relative standard deviation	45.5%
Median value	10.00	Relative error	4.9%

Analytical results in ascending order:

6	-20.00	O	22	9.30	10	12.00
36	0.01		11	10.00	24	13.00
26	5.00		23	10.00	37	13.20
3	7.00		1	10.00	2	20.44
5	7.39		21	10.40	9	20.50
27	8.45		19	10.90		
31	9.20		20	12.00		

O = Omitted result

Table 5.13. Statistics. Aluminium

Sample C

Analytical method: All
Unit: microg/L

Number of participants	22	Range	94
Number of omitted results	1	Variance	334
True value	178	Standard deviation	18
Mean value	176	Relative standard deviation	10.4%
Median value	178	Relative error	-1.4%

Analytical results in ascending order:

19	124	5	172	9	186	
35	151	33	178	29	189	
3	160	38	178	21	189	
20	163	1	180	24	192	
6	165	18	180	2	218	
22	170	37	182	28	382	O
4	171	13	184			
8	171	15	185			

O = Omitted result

Table 5.13. Statistics. Aluminium

Sample D

Analytical method: All
Unit: microg/L

Number of participants	22	Range	81
Number of omitted results	1	Variance	259
True value	157	Standard deviation	16
Mean value	157	Relative standard deviation	10.2%
Median value	157	Relative error	0.0%

Analytical results in ascending order:

19	115	8	156	24	165	
35	132	18	157	21	168	
3	143	1	157	37	169	
6	149	38	158	15	175	
20	149	33	161	2	196	
22	150	13	163	28	362	O
4	151	29	164			
5	156	9	164			

O = Omitted result

Table 5.14. Statistics. Iron
Sample C

Analytical method: All			
Unit: microg/L			
Number of participants	23	Range	36.18
Number of omitted results	1	Variance	42.34
True value	81.75	Standard deviation	6.51
Mean value	81.46	Relative standard deviation	8.0%
Median value	81.75	Relative error	-0.4%

Analytical results in ascending order:

29	63.82	35	80.66	4	83.90	
9	71.00	33	81.58	5	84.53	
8	77.64	38	81.70	13	84.90	
6	78.40	2	81.80	13	86.70	
20	79.10	32	81.94	24	87.00	
15	79.80	3	82.00	26	100.00	
21	80.20	37	82.20	19	110.00	O
1	80.30	22	83.00			

O = Omitted result

Table 5.14. Statistics. Iron

Sample D

Analytical method: All			
Unit: microg/L			
Number of participants	23	Range	45.92
Number of omitted results	1	Variance	102.15
True value	73.05	Standard deviation	10.11
Mean value	74.13	Relative standard deviation	13.6%
Median value	73.05	Relative error	1.5%

Analytical results in ascending order:

9	55.08	15	72.20	37	74.70	
8	61.10	32	72.70	5	75.42	
29	61.24	3	73.00	24	78.00	
6	69.70	1	73.10	19	80.00	O
20	70.10	22	74.00	26	85.00	
35	70.87	33	74.31	13	97.00	
2	71.60	4	74.40	13	101.00	
21	71.90	38	74.50			

O = Omitted result

Table 5.15. Statistics. Manganese

Sample C

Analytical method: All

Unit: microg/L

Number of participants	23	Range	9.08
Number of omitted results	0	Variance	3.56
True value	48.90	Standard deviation	1.89
Mean value	48.85	Relative standard deviation	3.9%
Median value	48.90	Relative error	-0.1%

Analytical results in ascending order:

35	45.02	1	48.20	21	49.40
6	46.30	13	48.60	9	49.70
20	46.90	4	48.80	26	49.93
32	47.06	13	48.90	2	51.03
8	47.21	3	49.00	5	51.04
18	47.89	22	49.00	38	51.10
19	48.00	24	49.00	37	54.10
15	48.10	29	49.37		

O = Omitted result

Table 5.15. Statistics. Manganese

Sample D

Analytical method: All

Unit: microg/L

Number of participants	23	Range	7.40
Number of omitted results	0	Variance	2.94
True value	43.00	Standard deviation	1.72
Mean value	43.00	Relative standard deviation	4.0%
Median value	43.00	Relative error	0.0%

Analytical results in ascending order:

35	39.40	9	42.61	13	43.80
6	40.60	13	43.00	22	44.00
32	40.93	3	43.00	24	44.00
26	41.11	19	43.00	5	44.63
20	41.50	15	43.20	2	45.20
8	41.69	4	43.30	37	45.80
18	42.05	29	43.30	38	46.80
1	42.60	21	43.60		

O = Omitted result

Table 5.16. Statistics. Cadmium

Sample C

Analytical method: All

Unit: microg/L

Number of participants	24	Range	1.99
Number of omitted results	2	Variance	0.28
True value	9.68	Standard deviation	0.53
Mean value	9.61	Relative standard deviation	5.5%
Median value	9.68	Relative error	-0.7%

Analytical results in ascending order:

26	5.95	O	15	9.45		22	9.80
29	7.46	O	6	9.55		21	9.80
35	8.71		1	9.60		5	9.81
20	8.81		18	9.62		2	9.90
3	8.90		38	9.66		24	10.00
32	8.94		37	9.70		4	10.40
19	9.07		9	9.70		33	10.43
8	9.13		13	9.76		12	10.70

O = Omitted result

Table 5.16. Statistics. Cadmium

Sample D

Analytical method: All

Unit: microg/L

Number of participants	24	Range	1.67
Number of omitted results	2	Variance	0.21
True value	8.59	Standard deviation	0.46
Mean value	8.48	Relative standard deviation	5.4%
Median value	8.59	Relative error	-1.3%

Analytical results in ascending order:

26	5.69	O	8	8.15		38	8.66
29	6.63	O	37	8.39		21	8.72
32	7.52		18	8.46		5	8.73
20	7.79		6	8.56		2	8.82
3	7.80		9	8.58		12	8.90
35	7.93		22	8.60		24	9.10
19	8.06		1	8.64		33	9.15
15	8.13		13	8.66		4	9.19

O = Omitted result

Table 5.17. Statistics. Lead

Sample C

Analytical method: All

Unit: microg/L

Number of participants	24	Range	2.26
Number of omitted results	2	Variance	0.37
True value	7.82	Standard deviation	0.61
Mean value	7.96	Relative standard deviation	7.6%
Median value	7.83	Relative error	1.8%

Analytical results in ascending order:

26	2.87	O	21	7.63	8	8.06
12	4.77	O	20	7.63	1	8.13
3	6.90		9	7.72	22	8.20
35	7.16		38	7.78	18	8.56
32	7.42		6	7.82	19	8.70
15	7.45		2	7.84	4	8.97
37	7.51		29	7.88	24	9.10
5	7.58		13	8.00	33	9.16

O = Omitted result

Table 5.17. Statistics. Lead

Sample D

Analytical method: All

Unit: microg/L

Number of participants	24	Range	2.70
Number of omitted results	2	Variance	0.30
True value	6.82	Standard deviation	0.55
Mean value	6.93	Relative standard deviation	8.0%
Median value	6.86	Relative error	1.7%

Analytical results in ascending order:

26	2.26	O	9	6.66	8	7.04
12	3.74	O	38	6.67	24	7.10
3	6.20		6	6.69	19	7.10
32	6.31		15	6.80	21	7.15
5	6.47		33	6.82	22	7.20
35	6.48		29	6.89	18	7.41
2	6.58		37	6.94	4	7.49
20	6.65		13	7.02	1	8.90

O = Omitted result

Table 5.18. Statistics. Copper

Sample C

Analytical method: All			
Unit: microg/L			
Number of participants	22	Range	7.76
Number of omitted results	0	Variance	2.42
True value	29.63	Standard deviation	1.56
Mean value	29.78	Relative standard deviation	5.2%
Median value	29.63	Relative error	0.5%

Analytical results in ascending order:

35	25.44	6	29.13	20	30.60
5	28.54	21	29.50	24	31.00
32	28.63	18	29.57	2	31.35
15	28.70	37	29.70	4	31.40
26	28.81	38	30.00	33	32.12
8	29.00	22	30.00	19	33.20
29	29.06	3	30.00		
1	29.10	13	30.40		

O = Omitted result

Table 5.18. Statistics. Copper

Sample D

Analytical method: All			
Unit: microg/L			
Number of participants	22	Range	6.72
Number of omitted results	0	Variance	1.93
True value	26.95	Standard deviation	1.39
Mean value	26.70	Relative standard deviation	5.2%
Median value	26.95	Relative error	-0.9%

Analytical results in ascending order:

35	22.78	6	26.35	13	27.40
26	24.53	18	26.70	4	27.90
29	25.78	38	26.90	24	28.00
5	25.81	1	27.00	33	28.05
8	25.82	22	27.00	2	28.29
15	26.00	3	27.00	19	29.50
32	26.05	37	27.10		
21	26.20	20	27.30		

O = Omitted result

Table 5.19. Statistics. Nickel

Sample C

Analytical method: All			
Unit: microg/L			
Number of participants	23	Range	3.10
Number of omitted results	0	Variance	0.54
True value	14.29	Standard deviation	0.73
Mean value	14.16	Relative standard deviation	5.2%
Median value	14.29	Relative error	-0.9%

Analytical results in ascending order:

15	12.30	1	14.00	37	14.60
32	13.00	22	14.00	21	14.70
3	13.20	6	14.01	4	14.70
35	13.53	29	14.29	13	14.80
18	13.59	38	14.40	24	15.00
20	13.60	33	14.43	5	15.18
8	13.90	19	14.50	9	15.40
26	13.97	2	14.52		

O = Omitted result

Table 5.19. Statistics.Nickel

Sample D

Analytical method: All			
Unit: microg/L			
Number of participants	23	Range	2.64
Number of omitted results	0	Variance	0.40
True value	12.74	Standard deviation	0.63
Mean value	12.59	Relative standard deviation	5.0%
Median value	12.74	Relative error	-1.1%

Analytical results in ascending order:

32	10.99	6	12.40	4	13.00
15	11.60	26	12.67	24	13.00
35	11.88	2	12.67	13	13.10
3	12.00	29	12.74	33	13.19
20	12.00	38	12.80	9	13.30
18	12.07	21	12.90	19	13.30
8	12.13	37	12.90	5	13.63
1	12.40	22	13.00		

O = Omitted result

Table 5.20. Statistics. Zinc

Sample C

Analytical method: All
Unit: microg/L

Number of participants	23	Range	3.92
Number of omitted results	1	Variance	0.78
True value	19.68	Standard deviation	0.88
Mean value	19.71	Relative standard deviation	4.5%
Median value	19.68	Relative error	0.2%

Analytical results in ascending order:

18	17.88	21	19.30	15	20.20	
3	18.50	13	19.30	2	20.38	
24	19.00	33	19.67	9	20.50	
6	19.05	5	19.68	35	20.57	
8	19.17	1	19.70	4	21.30	
37	19.20	29	19.88	20	21.80	
13	19.20	22	20.00	19	45.00	O
32	19.25	38	20.10			

O = Omitted result

Table 5.20. Statistics. Zinc

Sample D

Analytical method: All
Unit: microg/L

Number of participants	23	Range	4.01
Number of omitted results	1	Variance	0.83
True value	18.42	Standard deviation	0.91
Mean value	18.50	Relative standard deviation	4.9%
Median value	18.42	Relative error	0.4%

Analytical results in ascending order:

18	16.29	32	18.37	22	19.00	
35	16.96	13	18.40	24	19.00	
15	17.10	6	18.42	2	19.03	
3	18.00	8	18.42	9	19.30	
21	18.20	5	18.64	20	20.10	
37	18.30	29	18.68	4	20.30	
13	18.30	38	18.90	19	22.70	O
1	18.30	33	18.98			

O = Omitted result

Reports and publications from the ICP Waters programme

All reports from the ICP Waters programme from 2000 up to present are listed below. Reports before year 2000 can be listed on request. All reports are available from the Programme Centre. Reports and recent publications are also accessible through the ICP Waters website; http://www.icp-waters.no/

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Norsk institutt for vannforskning

Gaustadalléen 21 • 0349 Oslo
Telefon: 02348 • Faks: 22 18 52 00
www.niva.no • post@niva.no