Intercomparison 2236
pH, Conductivity, Alkalinity, NO3-N, Cl, SO3, Ca, Mg, Na, K, TOC, Tot-P, Tot-N, Al, Fe, Mn, Cd, Pb, Cu, Ni, and Zn.

Photo: Anne Luise Ribeiro

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Convention on Long-Range Transboundary Air Pollution

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Summary
Twenty-six laboratories from 15 countries accepted the invitation to join the ICP Waters chemical intercomparison and twenty-three of these successfully reported their results. Two sets of samples were prepared and distributed to the participants: one for the determination of ions and one for metals. In general, the results were of the same quality as previous years, with an overall acceptance rate of 75% (± 20% of the "true value", and for pH and conductivity ± 0.2 pH units and ± 10%, respectively). The highest acceptance rates (≥ 90%) were obtained for aluminium, iron and chloride. The lowest acceptance rates were for total phosphorous and total nitrogen, at 33%. Total nitrogen was included for the first time this year. General trends in the choice of techniques continue to shift towards plasma from atomic absorption, and to mass detection from ionic emission. This is especially promising for the determination of metals at low levels.

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CONVENTION OF LONG-RANGE
TRANSBOUNDARY AIR POLLUTION

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

Intercomparison 2236
pH, Conductivity, Alkalinity, NO$_3$-N, Cl, SO$_4$, Ca, Mg, Na, K, TOC, Total-P, Total-N, Al, Fe, Mn, Cd, Pb, Cu, Ni, and Zn

Prepared at the ICP Waters Programme Centre
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Preface

The International Cooperative Programme on Assessment and Monitoring of the 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 (CLRTAP) in July 1985. Since then, ICP Waters has been an important contributor to document the effects of implementing the Protocols under the Convention. ICP Waters has prepared numerous assessments, reports and publications that address the effects of long-range transported air pollution.

ICP Waters and its Programme Centre is chaired and hosted by the Norwegian Institute for Water Research (NIVA). A programme subcentre is established at NORCE, Bergen. ICP Waters is supported financially by the Norwegian Ministry of Climate and Environment and the Trust Fund of the UNECE LRTAP Convention.

The main aim of the ICP Waters programme is to assess, on a regional basis, the degree and geographical extent of the impact of atmospheric pollution, in particular acidification, on surface waters. More than 20 countries in Europe and North America participate in the programme on a regular basis.

An objective of the ICP Waters programme is to establish and maintain an international network of surface water monitoring sites and to promote international harmonisation of monitoring practices. A tool in this work is inter-laboratory quality assurance tests. Here biases between analyses carried out by the individual participants of the programme are identified and controlled.

Here we report the results from the 36th intercomparison of chemical analyses.

Oslo, December 2022

Tina Bryntesen
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Summary

The chemical interlaboratory comparison is an important tool for the ICP Waters to ensure consistency and comparability of the surface water monitoring results among the programme participants. The test is conducted yearly and is based on the "round robin" principle. In short, the same water sample is distributed to all the participating laboratories which analyse the sample for a set repertoire of parameters with their method of choice. Then, the results are compiled and analysed using the Youden test statistic. The "true value" for each parameter is calculated as the median of the reported results after excluding extreme observations. Two different sets of samples are prepared and distributed, one for the determination of ions and the other for metals.

The 2236 edition of the test was conducted in the period from May to October 2022. A total of 26 laboratories representing 15 different countries signed up. Among these, 23 laboratories representing 15 countries successfully reported results to the database. The participants were invited to determine pH, conductivity, alkalinity, nitrate+nitrite nitrogen, chloride, sulphate, calcium, magnesium, sodium, potassium, total organic carbon, total phosphorus, total nitrogen, aluminium, iron, manganese, cadmium, lead, copper, nickel, and zinc. The acceptance limit was typically at ± 20% of the "true value", except for pH and conductivity (± 0.2 pH units and ± 10%, respectively).

Overall, the acceptance rates for the 2236 edition were similar to the rates from recent years. 75% of the results were within the target threshold (an acceptance rate calculated from all the reported results). Several different factors can influence the acceptance rate, such as the concentration of the analyte in the sample and the choice of analytical techniques among the laboratories. This year, the concentrations of many analytes were relatively low, which likely explains some of the lower acceptance rates.

The highest acceptances were obtained for aluminium (100%), iron (100%) and chloride (90%). Parameters which had acceptance rates between 80-89% were magnesium, sodium, potassium, total organic carbon, manganese, cadmium, lead, copper, and zinc. The poorest acceptance rates were obtained for total phosphorous and total nitrogen, both with 33% acceptable results. Many participants reported that the concentration levels were around or below their quantification limits.

For several of the parameters, different analytical techniques had been used by the various laboratories. The use of different techniques can challenge the unity of the results, with the effect typically being more severe for low analyte concentrations. For several of the ions, up to five different techniques had been used, while for the metals the number of different techniques was three to four. Some overall patterns in the preferred technique could be found: Ion chromatography was preferred for the determination of the negatively charged ions. For the positively charged ions, it was most common to use either ion chromatography (IC) or some form of inductively coupled plasma technique - optical emission spectroscopy (ICP-OES) or mass spectrometry (ICP-MS). For all the metals, the sensitive ICP-MS was the preferred technique of choice. This confirms the trends observed in the last years, that plasma techniques are taking over for the more traditional atomic absorption techniques, and that the much more sensitive mass detector is replacing the optical emission spectroscopy detector.

1   Introduction

The international cooperative programme for assessment and monitoring of the effects of air pollution on rivers and lakes (ICP Waters) works to assess the degree to which atmospheric pollution has affected surface waters. The programme was established in 1985 under the Executive Body of the United Nations Economic Commission for Europe (UNECE). The Focal Centres in each country contributes with data from their national monitoring programmes.

To ensure that the results across the entire ICP Waters are consistent and comparable, inter-laboratory quality controls are necessary, as stated in the "ICP Waters Programme Manual" (1). In a multi laboratory programme, typical causes of inconsistency include the use of different types of analytical techniques, errors in the calibration procedure, etc. The between-laboratory control carried out by the Programme Centre of ICP Waters is based on the "round robin" concept meaning that the same sample is analysed by the different participating laboratories using their analytical principle and method of choice. The analytical results are analysed using the Youden test statistics (2, 3) that assesses the consistency of the results between the laboratories, and can also indicate whether the results are affected by a systematic effect (e.g. different analytical techniques give slightly different results) or only by random errors (typically more profound at levels close to the limit of quantification). The Youden test is briefly described in Appendix C. The levels of the variables should be set to be as close to the expected natural levels as possible, and that the range from year-to-year shall cover the variation among countries of the participating laboratories.

Several factors can contribute to the acceptance ratio and these should be considered when evaluating the results, and when considering measures to improve the results from individual laboratories. For example, different methods used by different laboratories may give systematically different results (higher or lower). Based on the method used by most of the participating laboratories, the "true value" may be biased. Such systematic effect will be evident in the distribution of the results in the Youden chart, by the points residing along the 45° angled line. One other cause of poor acceptance ratio is when the concentration in the sample is low, and close to the limit of quantification of the method used. Then the effect from random errors can be problematic. This will most often appear in the Youden chart as a distribution of the results perpendicular to the 45° angled line.

This thirty-sixth chemical intercomparison test, called 2236, covered the determination of the following constituents of natural surface waters: pH, conductivity, alkalinity, nitrate+nitrite nitrogen, chloride, sulphate, calcium, magnesium, sodium, potassium, total organic carbon, total phosphorus, total nitrogen, aluminium, iron, manganese, cadmium, lead, copper, nickel, and zinc. While most of these variables have been part of the test since it started, total organic carbon and aluminium was included in 2009, and total phosphorus in 2017. This year, total nitrogen was also included for the first time.

2   Procedures of the intercomparison

Two different sets of samples were prepared by the Programme Centre and distributed to the participating laboratories: one pair for the determination of major ions and one pair for the determination of metals (as agreed upon at the Task Force meeting in Burlington, Canada, 2009). The procedure for the preparation of the two sample sets is presented in Appendix B.

The samples were shipped from the Programme Centre during week 26. Unfortunately, several participants experienced delay and/or were prompted to pay taxes for the shipment. At least one participant opted out of reporting their results, due to a prolonged delay of the delivery. Participants were encouraged to conduct the analyses as soon as possible after reception.

As mentioned, the analytical results were treated by the Youden method (2, 3) to evaluate the comparability of the analytical results produced by the laboratories participating in the International Cooperative Programme, and to assess potential systematic and/or random error in the distribution of the results. For each variable, the "true value" was calculated as the median of the reported results, after excluding extreme observations. This way of setting the "true value" is considered acceptable if the participants mainly use the same analytical techniques. However, this is not always the case, and for parameters such as pH and alkalinity different techniques/methods are frequently used and producing strong systematic bias in the results. Since not one method can be argued to be better than another, this issue has persisted in the interlaboratory harmonisation.

The criteria for acceptable results were for most variables ± 20% of the "true value", as outlined in the Manual for Chemical and Biological Monitoring (1). Exceptions from this were pH and conductivity, for which the acceptance limits were set to ± 0.2 pH units and ± 10%, respectively. This year, there is also an exception for nitrate-nitrite nitrogen, where only sample B results will be discussed and evaluated. The reason for this was a discrepancy between the level in the two samples, likely resulting from biological transformation processes in one of the two.

3   Results and Discussion

In the 2236 edition of the chemical intercomparison test, a total of 26 laboratories (representing 15 different countries) registered to participate, and 23 of the laboratories representing 15 different countries successfully registered their results. Information about the participating laboratories is provided in Appendix A, both by the identity of the laboratories (Table A.1.) and by a summary of the different countries represented (Table A.2.).

In Table 1, the results from the 2236 chemical intercomparison test is summarised, constituting for each parameter: the number of participants, the acceptance ratio, and for comparison the acceptance ratio from the three previous years. Overall, the results were quite similar to previous years, with a summarized acceptance ratio indicating that 75% of the results were within the general target accuracy of 20%, or the special accuracy limit for pH and conductivity (± 0.2 pH units and ± 10% respectively.

Throughout this chapter the results for each variable will be presented and discussed based on acceptance ratio (Table 1) and the visual distribution of the results in the Youden chart (Figures 1-21). In the Youden chart, each laboratory is presented by one point, and the distribution of points can indicate the occurrence of random and/or systematic errors among the laboratories. The acceptance limit (typically ± 20 % of the mean true values for the sample pair) is illustrated in the charts as a circle. Note that laboratories with results that strongly deviated from the others has been excluded from the charts. Information on the different analytical techniques used by the laboratories is shown in Table 2. Factors that are typically found to influence the compliance among the results are low parameter values, the use of several different analytical methods for the determination of the same parameter, both leading to increased variability in the results.

For more detailed information on the uncertainty of the "true values" see Table C.1. (Appendix C). The calculation has been performed according to ISO 13528 (2022), "Statistical methods for use in proficiency testing by interlaboratory comparisons". The individual results reported by the laboratories are listed in Table D.1. (Appendix D), and more detailed statistics for each parameter is presented in Tables D.2.1 to D.2.21 (Appendix D).

Table 1. Summary of the results including the true values, number of participating laboratories, and acceptance rate in the 2236 edition and the three previous years (2135, 2034, and 1933) for each parameter.

Parameter (unit)	Sample-pair	True value Sample 1	True value Sample 2	Acceptable limit, %	Number of pairs Tot.	Number of pairs Accept.	Acceptable results for intercalibration (%) 2236	2135	2034	1933
pH	AB	6.17	6.21	0.2 pH	22	11	50	65	75	60
Conductivity (mS/m)	AB	1.90	1.77	10	21	12	57	64	80	79
Alkalinity (mmol/L)	AB	0.070	0.070	20	14	8	57	42	44	62
Nitrate + nitrite-nitrogen (µg N/L)	B	-	25.0	20	19*	9*	47	50	47	69
Chloride (mg/L)	AB	1.14	0.96	20	20	18	90	83	90	93
Sulphate (mg/L)	AB	1.31	1.19	20	21	16	76	91	76	75
Calcium (mg/L)	AB	1.75	1.63	20	28	22	79	69	89	90
Magnesium (mg/L)	AB	0.390	0.353	20	28	25	89	71	95	93
Sodium (mg/L)	AB	1.14	1.00	20	27	23	85	89	100	96
Potassium (mg/L)	AB	0.258	0.220	20	27	22	81	74	95	85
Total organic carbon (mg/L)	AB	7.15	6.49	20	16	13	81	71	73	80
Total phosphorous (µg P/L)	AB	10.9	9.1	20	18	6	33	29	41	35
Total nitrogen (µg N/L)	AB	278	258	20	12	4	33	-	-	-
Aluminium (µg/L)	CD	151	141	20	17	17	100	67	80	55
Iron (µg/L)	CD	120	110	20	21	21	100	56	94	76
Manganese (µg/L)	CD	2.29	2.21	20	16	13	81	65	93	71
Cadmium (µg/L)	CD	0.650	0.550	20	16	14	88	56	94	77
Lead (µg/L)	CD	2.75	2.65	20	17	14	82	24	88	73
Copper (µg/L)	CD	3.10	4.48	20	15	13	87	70	94	75
Nickel (µg/L)	CD	1.89	1.60	20	17	13	76	72	94	77
Zinc (µg/L)	CD	10.6	11.9	20	19	16	84	72	80	61
Total					411	310	75	(65)	(81)	(75)

* Number of results instead of number of pairs.

3.1   pH

Values of pH were reported by 22 laboratories, among which 50% were within the acceptable limit (± 0.2 pH units of the "true value", Table 1). Over several years, pH has been associated with poor acceptance ratio and this has been attributed to the use of different measuring methods. E.g. the different practices of stirring or not stirring the sample during determination can give a systematic error, and this is especially the case for samples with lower total ionic strength (4, 5). This year, the number of laboratories using each of the different methods was relatively evenly distributed (Table 2). The most used method was electrometric determination with stirring (10 laboratories), followed by electrometric with equilibration (6 laboratories) and determination without stirring (4 laboratories). The last 2 participants reported to have used an unspecified method. The Youden chart showed that systematic error dominates the distribution of the results for pH (Figure 1). The results measured without stirring are mostly in the higher end of the reported results, so the median results are likely biased towards stirring rather than non-stirring.

It is important to remember that pH is a very sensitive parameter to determine, and that sample storage and handling, as well as the use of different analytical techniques can affect the results. This parameter should be determined as soon as possible after the samples have arrived at the laboratory.

3.2   Conductivity

In this 2236 edition, conductivity was measured by 21 laboratories and showed an acceptance rate of 57%. This is a low acceptance rate for conductivity, but similar to last year. In both years the samples had relatively low conductivity. As the acceptance limit is set at 10% for this parameter, small variations will lead to results being outside of the acceptance limit. The result is also biased due to two result pairs (corresponding to almost 10% of the results) seemingly reported using wrong units.

Of the participants, 19 reported to have used electrometry for the determination of conductivity while 2 reported to have used an unspecified method (Table 2). The Youden chart (Figure 2) shows that random errors dominate the distribution of the results. Conductivity is highly temperature dependent, and improper temperature correction may lead to deviating results. Conductivity will vary by 2% for each degree at the temperatures around room temperature.

3.3   Alkalinity

Alkalinity was reported by 14 of the participating laboratories, producing an acceptance ratio of 57% (± 20% of the "true value", Table 1). Gran plot titration method, which is the suggested reference method in the manual (1) was used by 5 laboratories, and 4 laboratories used two end points titration. Of the rest, 3 reported to have used one end point titration, but the end point differed between pH 4.5, 5.6, and an unspecified pH. The last 2 participants reported to have used colorimetry and an unspecified method.

The Youden chart (Figure 3) shows that the results are distributed along the 45° line, indicating that most of the errors are systematic. It is also worth noting that there is a separate grouping of results outside the Youden diagram, from labs having reported results around 0.110 to 0.130 mmol/L. 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 = 4.5. In such case, the relative error introduced by assuming affixed end-point pH of 4.5, 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. This is likely true for the high grouping of results, and laboratories using these methods must evaluate their results with caution.

3.4   Nitrate + nitrite-nitrogen

Concentrations of nitrate+nitrite nitrogen was reported by 19 laboratories. Unfortunately, it seems something happened to the A sample, as over half of the participants reported the result as zero or below LOQ. During the preparation of the sample set, the level of nitrate+nitrite nitrogen was measured to be similar in samples A and B (35 and 31 µg N/l), and we thus anticipate that the conditions in sample A has been favourable for transformation processes.

Due to limitations in the statistic generation of the intercomparison test, only sample pairs with two accepted results are normally counted towards the "true value". Calculations were therefore done manually, in order to get a fair estimation of the "true value" in sample B. The acceptance limit was set to 20%, and the results are shown in a graph in Figure 4. This resulted in 47% of the results being deemed acceptable.

3.5   Chloride

For chloride an acceptance rate of 90% (± 20% of the "true value") was achieved by the 20 participating laboratories (Table 1). This is a good result, especially with the relatively low concentration of chloride in the samples. According to Table 2, ion chromatography was the technique of choice by most of the participants (18 laboratories). The last 2 participants had used photometry with autoanalyzer and electrometry.

The distribution of the results in the Youden diagram (Figure 5) shows that random error is the dominating source of error, and many results have a very small amount of error.

3.6   Sulphate

Results for sulphate was reported by 21 laboratories, producing an acceptance rate at 76% (± 20% of the "true value", Table 1). This is lower than last year, but it is similar to the years before. The preferred technique for sulphate determination was ion chromatography (18 participants), followed by ICP-OES (3 participants).

The Youden chart in Figure 6 shows that most errors are small and systematic.

3.7   Calcium

The acceptance rate for calcium was around average of the last years, at 79% (± 20% of the "true value", Table 1), for the 28 reporting laboratories. The acceptance rate seems to follow the concentration of the parameter.

The different techniques that had been used for the determination of calcium (Table 2) constituted ion chromatography (10 laboratories), ICP-OES (9 laboratories), ICP-MS (6 laboratories) and flame atomic absorption spectrometry (FAAS) (2 laboratories). The final participant reported to have used an unspecified method. The Youden diagram in Figure 7 shows that the dominating errors are systematic.

3.8   Magnesium

Levels of magnesium was reported by 28 laboratories. The acceptance ratio was 89%, which is a normal level for this parameter. The different techniques and methods that had been used for the determination of magnesium are listed in Table 2, and constituted ion chromatography (10 laboratories), ICP-OES (8 laboratories), ICP-MS (7 laboratories), FAAS (2 laboratories), and an unspecified method (1 laboratory).

The Youden diagram in Figure 8 shows that many participants have reported very precise results, and the distribution of the rest of the results is dominated by systematic errors.

3.9   Sodium

An acceptance rate of 85% was achieved for sodium this year, and results were provided by 27 laboratories. Sodium has typically showed high acceptance rates during the previous years. Five different techniques had been used by the laboratories for the determination of sodium: Ion chromatography (10 laboratories), ICP-OES (8 laboratories), ICP-MS (6 laboratories), FAAS (2 laboratories), and an unspecified method (1 laboratory).

The good agreement of the sodium concentrations between the laboratories was confirmed by the distribution in the Youden chart, showing only a small, mostly systematic, variation in the results (Figure 9).

3.10   Potassium

For potassium, 27 laboratories reported results from which 81% were within the acceptable threshold (± 20% of the "true value", Table 1). This is an acceptance rate which is around average of the last years. Five different techniques had been used by the laboratories for the determination of potassium: Ion chromatography (10 laboratories), ICP-OES (8 laboratories), ICP-MS (6 laboratories), FAAS (2 laboratories), and an unspecified method (1 laboratory).

The Youden diagram in Figure 10 shows that the spread of the results is a mix of both systematic and random errors.

3.11   Total organic carbon

Concentrations of total organic carbon was reported by 16 laboratories, among which 81% were within the target threshold (± 20% of the "true value", Table 1). This was comparable to the results from the previous years.

Most of the laboratories (13 laboratories) had used the technique of combustion for the determination of total organic carbon, while 3 laboratories had used the UV/peroxodisulphate technique, and 1 laboratory had used an unspecified method. There was no apparent bias in the results depending on the method used for analysis. The Youden chart for total organic carbon showed a mix of both systematic and random errors in the distribution of the results (Figure 11).

3.12   Total phosphorus

Total phosphorus was reported by 18 laboratories (Table 1). The acceptance rate was one of the lowest among the parameters this year, at 33%. The acceptance rate of this parameter has been low since it was included in the chemical intercomparison (in 2017). Several participants have reported that the results were below their LOQ.

According to Table 2, most participants used photometry for the determination of total phosphorus (9 participants), followed by ICP-OES (4 participants). Ion chromatography was used by 2 participants, ICP-MS was used by one, and the final two reported to have used an unspecified method. The Youden chart (Figure 12) is showing a large spread, with random errors within the 20% circle and some results which are systematically high/low. It is noted that for next year, we should aim for a higher concentration of phosphorus, in order to get results farther away from participants' LOQ. With a large spread in results, the uncertainty of the "true values" are quite high.

3.13 Total nitrogen

Total nitrogen was included for the first time this year, and it was reported by 12 laboratories (Table 1). The acceptance rate was only 33%. A few laboratories marked the results as being below or near their LOQ.

Several methods were reportedly used; EN 12260 (4 laboratories), EN ISO 11905 (3 laboratories), EN ISO 20236 (2 laboratories), and Inline-digestion CFA (1 laboratory). In addition, 2 laboratories reported to have used an unspecified method. EN 12260 is a withdrawn standard method which has been replaced by EN ISO 20236, and these cover determination after catalytic oxidative combustion. EN ISO 11905 is determination after oxidation in alkaline environment using peroxodisulphate.

The Youden chart (Figure 13) shows that the reported results are spread over a large range, and there is a high degree of uncertainty of the "true value". The water contained a relatively high amount of organic carbon, and a relatively large amount of the nitrogen will likely also be organically bound, which again is harder to determine than inorganic carbon.

3.14 Aluminium

Concentrations of aluminium were reported by 17 laboratories, producing an acceptance rate at 100% (± 20% of the "true value", Table 1). This is higher than the previous years, where the average has been around 70%. The increase is most likely explained by the concentration levels, which now were three times higher than last year.

Three techniques were used for the determination of aluminium (Table 2): ICP-MS (11 laboratories), ICP-OES (5 laboratories), and graphite furnace atomic absorption spectrometry (GFAAS) (1 laboratory). The Youden chart for aluminium (Figure 14) shows that most of the errors were small and systematic.

3.15 Iron

Results reported for iron showed an acceptance ratio at 100% for the 21 reporting laboratories (± 20% of the "true value", Table 1). Like aluminium, the acceptance ratio follows the concentration levels.

Four techniques were used for the determination of iron (Table 2), constituting ICP-MS (11 laboratories), ICP-OES (8 laboratories), FAAS (1 laboratory), and photometry (1 laboratory). The Youden chart (Figure 15) shows that the spread of the results is mostly small and systematic, being similar to aluminium.

3.16 Manganese

The acceptance rate for manganese was at 81% for the 16 laboratories providing results (± 20% of the "true value", Table 1). This is higher than last year, which is slightly surprising as the concentration levels are similar to last year.

For the determination of manganese, 10 laboratories had used ICP-MS, 5 had used ICP-OES and the last laboratory had used FAAS (Table 2). The Youden chart in Figure 16 showed mostly small random errors.

3.17 Cadmium

Cadmium was determined by 16 of the participating laboratories, providing results with an acceptance rate of 88% (± 20% of the "true value", Table 1). This is better than last year, especially considering the concentration levels being very similar.

ICP-MS was the determination method used by most of the participants (12 laboratories), followed by GFAAS and ICP-OES (2 laboratories each). The Youden chart (Figure 17) shows mostly random errors.

3.18 Lead

Lead was reported by 17 laboratories producing an acceptance rate of 82%. This is around the average acceptance rate for this parameter.

According to Table 2, the most used method for determination of lead was ICP-MS (12 laboratories), followed by ICP-OES and GFAAS (3 and 2 laboratories, respectively). Looking at the Youden chart (Figure 18), the results are mostly small and systematic

3.19 Copper

The acceptance rate for copper was at 87% for the results provided by 15 laboratories (± 20% of the "true value", Table 1). This was comparable to the results from the previous years. For determination, 11 participants had used ICP-MS, 3 had used ICP-OES and the last participant had used FAAS.

The distribution of the results in the Youden chart in Figure 19 shows that small random errors dominate the results.

3.20 Nickel

Results for nickel was reported by 17 laboratories for which 76% were classified as acceptable according to the target limit (± 20% of the "true value", Table 1). For its determination, 12 participants had used ICP-MS, 3 had used ICP-OES and the last 2 participants had used GFAAS. The Youden chart (Figure 20) shows that systematic errors are dominating the spread of the results.

3.21 Zinc

Concentration of zinc in the samples were determined by 19 laboratories from which 84% fulfilled the acceptance criteria (± 20% of the "true value", Table 1). This is similar to the previous years.

For determination, 13 participants had used ICP-MS, 5 had used ICP-OES and the last participant had used FAAS. The Youden chart in Figure 21 shows that systematic errors dominate the results.

Table 2. Statistical summary of the results from the 2236 edition, including information of the different analytical techniques used by the laboratories.

Parameter and method	Sample pair	True value S1	True value S2	No. Lab Total	No. Lab O	Median S1	Median S2	Sample 1 Avg.	Sample 1 SD	Sample 2 Avg.	Sample 2 SD	Rel. SD % S1	Rel. SD % S2	Rel. error % S1	Rel. error % S2
pH	AB	6.17	6.21	22	0	6.17	6.21	6.22	0.24	6.25	0.20	3.8	3.2	0.7	0.6
Elec., stirring				10	0	6.16	6.19	6.14	0.24	6.19	0.19	3.9	3.0	-0.6	-0.4
Elec., equilibration				6	0	6.13	6.21	6.22	0.21	6.23	0.18	3.3	2.9	0.8	0.4
Elec., non-stirring				4	0	6.44	6.46	6.39	0.28	6.41	0.25	4.3	3.9	3.6	3.2
Other method				2	0			6.26		6.25				1.5	0.6
Conductivity	AB	1.90	1.77	21	2	1.90	1.77	1.85	0.17	1.73	0.14	9.1	8.2	-2.8	-2.4
Electrometry				19	1	1.91	1.78	1.87	0.15	1.74	0.14	7.9	7.9	-1.7	-1.8
Other method				2	1			1.48		1.54				-22.1	-12.9
Alkalinity	AB	0.070	0.070	14	4	0.070	0.070	0.072	0.007	0.071	0.006	9.4	8.3	2.8	2.1
Gran plot titration				5	1	0.079	0.078	0.079	0.006	0.076	0.007	7.6	8.7	12.1	8.9
Two end points titration				4	0	0.068	0.068	0.068	0.002	0.068	0.003	3.0	4.7	-2.7	-2.6
Colorimetry				1	0			0.066		0.069				-5.7	-1.4
One end point(other)titr.				1	1			0.130		0.130				85.7	85.7
One end point(pH4.5)titr				1	1			0.112		0.114				60.0	62.9
One end point(pH5.6)titr.				1	0			0.067		0.068				-4.3	-2.9
Other method				1	1			0.120		0.113				71.4	61.4
Nitrate + nitrite-nitrogen	B		25.0	19	4		25.0	-	-	23.9	5.8		24.0		
Ion chromatography				15	4										
Auto.,photometry, Cd red				3											
Photometry, other method				1											
Chloride	AB	1.14	0.96	20	2	1.14	0.96	1.13	0.07	0.94	0.06	6.0	6.1	-0.5	-2.5
Ion chromatography				18	1	1.14	0.95	1.14	0.07	0.93	0.06	6.1	6.1	-0.3	-2.8
Electrometry				1	0			1.09		0.99				-4.4	3.5
Photometry, autoanalyzer				1	1			0.36		0.16				-68.2	-83.8
Sulphate	AB	1.31	1.19	21	1	1.31	1.19	1.29	0.18	1.18	0.15	13.9	12.3	-1.2	-0.8
Ion chromatography				18	0	1.30	1.18	1.26	0.16	1.15	0.13	12.3	10.9	-3.8	-3.1
ICP-OES				3	1			1.60		1.43				22.1	19.8
Calcium	AB	1.75	1.63	28	1	1.75	1.63	1.77	0.20	1.62	0.15	11.3	9.5	1.0	-0.8
Ion chromatography				10	1	1.73	1.64	1.71	0.27	1.61	0.21	15.6	13.1	-2.5	-1.1
ICP-OES				9	0	1.73	1.63	1.80	0.17	1.63	0.12	9.4	7.2	2.8	0.1
ICP-MS				6	0	1.88	1.66	1.88	0.05	1.66	0.07	2.8	4.2	7.2	2.1
FAAS				2	0			1.58		1.43				-10.0	-12.3
Other method				1	0			1.79		1.61				2.2	-1.1
Magnesium	AB	0.390	0.353	28	1	0.390	0.353	0.387	0.031	0.351	0.024	7.9	6.9	-0.8	-0.7
Ion chromatography				10	1	0.380	0.351	0.381	0.039	0.351	0.030	10.2	8.7	-2.4	-0.6
ICP-OES				8	0	0.378	0.348	0.376	0.025	0.342	0.026	6.5	7.7	-3.7	-3.3
ICP-MS				7	0	0.399	0.356	0.409	0.022	0.359	0.016	5.4	4.6	4.8	1.7
FAAS				2	0			0.386		0.353				-1.2	-0.1
Other method				1	0			0.388		0.357				-0.5	1.1
Sodium	AB	1.14	1.00	27	3	1.14	1.00	1.13	0.08	0.99	0.07	7.0	6.7	-0.7	-1.3
Ion chromatography				10	0	1.14	1.00	1.15	0.05	1.01	0.04	4.0	4.2	1.2	0.9
ICP-OES				8	2	1.10	1.00	1.12	0.07	0.98	0.07	6.6	7.3	-2.1	-1.9
ICP-MS				6	1	1.14	0.97	1.14	0.06	0.99	0.04	5.5	4.3	-0.3	-1.4
FAAS				2	0			1.18		1.00				3.4	0.1
Other method				1	0			0.91		0.78				-20.4	-21.6

Table 2. cont.

Parameter and method	Sample pair	True value S1	True value S2	No. Lab Total	No. Lab O	Median S1	Median S2	Sample 1 Avg.	Sample 1 SD	Sample 2 Avg.	Sample 2 SD	Rel. SD % S1	Rel. SD % S2	Rel. error % S1	Rel. error % S2
Potassium	AB	0.258	0.220	27	4	0.258	0.220	0.255	0.019	0.218	0.014	7.6	6.5	-1.1	-1.1
Ion chromatography				10	1	0.254	0.220	0.249	0.022	0.213	0.010	8.9	4.9	-3.3	-3.0
ICP-OES				8	2	0.258	0.214	0.251	0.018	0.217	0.023	7.1	10.5	-2.6	-1.3
ICP-MS				6	0	0.271	0.226	0.264	0.018	0.223	0.010	6.8	4.6	2.4	1.1
FAAS				2	1			0.260		0.220				0.8	0.0
Other method				1	0			0.272		0.227				5.4	3.2
Total organic carbon	AB	7.15	6.49	16	0	7.15	6.49	7.21	0.85	6.57	0.64	11.7	9.8	0.8	1.2
Combustion				12	0	6.97	6.44	7.14	0.93	6.49	0.58	13.0	9.0	-0.1	0.0
UV/peroxodisulphate				3	0	7.47	6.77	7.65	0.46	7.08	0.78	6.0	11.1	7.0	9.1
Other method				1	0			6.70		5.97				-6.3	-8.0
Total phosphorous	AB	10.9	9.1	18	8	10.9	9.1	10.8	2.8	9.1	2.6	26.2	28.8	-0.6	0.4
Photometry				9	2	11.0	9.0	11.1	2.5	9.2	2.7	23.0	29.7	1.6	0.6
ICP-OES				4	1	10.9	9.3	10.3	4.0	9.1	3.0	39.2	33.0	-5.7	0.0
Ion chromatography				2	2			0.0		1.9				-100	-79
Other method				2	2			21.4		11.7				96	28
ICP-MS				1	1			33.0		26.0				203	186
Total nitrogen	AB	278	258	12	0	278	258	293	57	262	48	19.4	18.2	5.3	1.7
EN 12260				4	0	250	211	241	34	247	75	14.2	30.5	-13.2	-4.2
EN ISO 11905				3	0	270	260	301	74	265	42	24.7	15.9	8.1	2.6
EN ISO 20236				2	0			303		257				9.0	-0.4
Other method				2	0			352		299				26.6	15.7
Inline-digestion CFA				1	0			337		256				21.2	-0.8
Aluminium	CD	151	141	17	0	151	141	151	7	141	7	4.5	4.9	0.2	0.0
ICP-MS				11	0	150	140	149	5	138	6	3.3	4.0	-1.6	-1.8
ICP-OES				5	0	153	142	155	8	145	7	4.8	4.9	2.7	2.6
GFAAS				1	0			162		152				7.4	7.7
Iron	CD	120	110	21	0	120	110	118	6	107	6	5.0	5.3	-1.8	-2.4
ICP-MS				11	0	120	107	117	7	106	7	5.6	6.2	-2.9	-3.6
ICP-OES				8	0	120	110	119	5	109	4	3.8	3.3	-0.7	-1.1
FAAS				1	0			114		102				-5.3	-7.6
Photometry				1	0			126		116				4.8	5.0
Manganese	CD	2.29	2.21	16	1	2.29	2.21	2.22	0.19	2.15	0.20	8.4	9.1	-3.1	-2.8
ICP-MS				10	0	2.23	2.16	2.16	0.20	2.09	0.22	9.3	10.4	-5.6	-5.2
ICP-OES				5	0	2.30	2.22	2.33	0.07	2.25	0.08	3.1	3.5	1.8	2.0
FAAS				1	1			3.20		3.40				39.7	53.8
Cadmium	CD	0.650	0.550	16	1	0.650	0.550	0.640	0.047	0.555	0.032	7.3	5.7	-1.6	0.9
ICP-MS				12	1	0.660	0.550	0.659	0.024	0.560	0.023	3.6	4.1	1.5	1.9
GFAAS				2	0			0.543		0.499				-16.5	-9.3
ICP-OES				2	0			0.627		0.582				-3.5	5.8
Lead	CD	2.75	2.65	17	1	2.75	2.65	2.77	0.34	2.66	0.30	12.3	11.4	0.8	0.5
ICP-MS				12	0	2.77	2.69	2.80	0.10	2.68	0.12	3.5	4.5	1.9	1.1
ICP-OES				3	1			2.78		2.93				1.1	10.6
GFAAS				2	0			2.59		2.31				-5.8	-13.0

Table 2. cont.

Parameter and method	Sample pair	True value		No. Lab		Median		Sample 1		Sample 2		Rel. SD %		Rel. error %	
		S1	S2	Total	O	S1	S2	Avg.	SD	Avg.	SD	S1	S2	S1	S2
Copper	CD	3.10	4.48	15	0	3.10	4.48	3.17	0.41	4.50	0.26	13.0	5.7	2.3	0.5
ICP-MS				11	0	3.10	4.48	3.03	0.14	4.46	0.20	4.5	4.5	-2.1	-0.4
ICP-OES				3	0	3.14	4.83	3.37	0.71	4.70	0.44	21.2	9.5	8.7	4.8
FAAS				1	0			4.10		4.40				32.3	-1.8
Nickel	CD	1.89	1.60	17	1	1.89	1.60	1.87	0.23	1.63	0.24	12.1	14.7	-0.9	1.6
ICP-MS				12	1	1.88	1.60	1.88	0.11	1.61	0.11	5.7	7.0	-0.7	0.9
ICP-OES				3	0	1.90	1.95	2.02	0.23	1.90	0.23	11.4	12.1	7.1	18.7
GFAAS				2	0			1.63		1.28				-13.8	-20.3
Zinc	CD	10.6	11.9	19	1	10.6	11.9	10.5	1.0	11.9	1.1	9.5	8.8	-0.5	-0.1
ICP-MS				13	1	11.0	12.4	11.0	0.7	12.3	0.8	6.5	6.1	3.5	3.4
ICP-OES				5	0	10.2	11.5	9.7	1.1	11.2	1.2	11.7	10.5	-8.4	-5.6
FAAS				1	0			9.6		10.2				-9.4	-14.3

pH
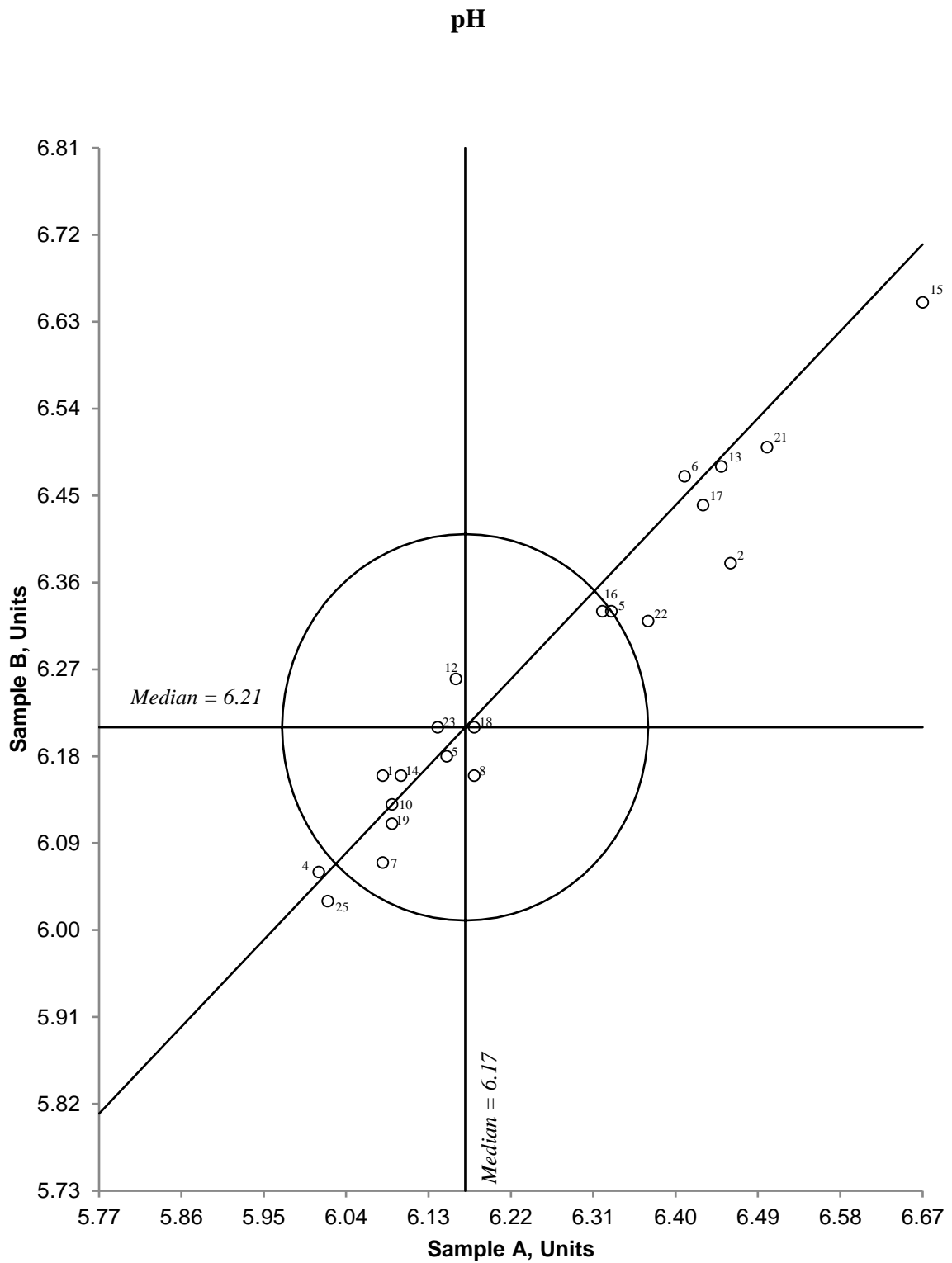
Figure 1. Youden diagram for pH. Sample pair AB. Acceptance limit, given by circle, is 0.2 pH units (3.23%).

Conductivity
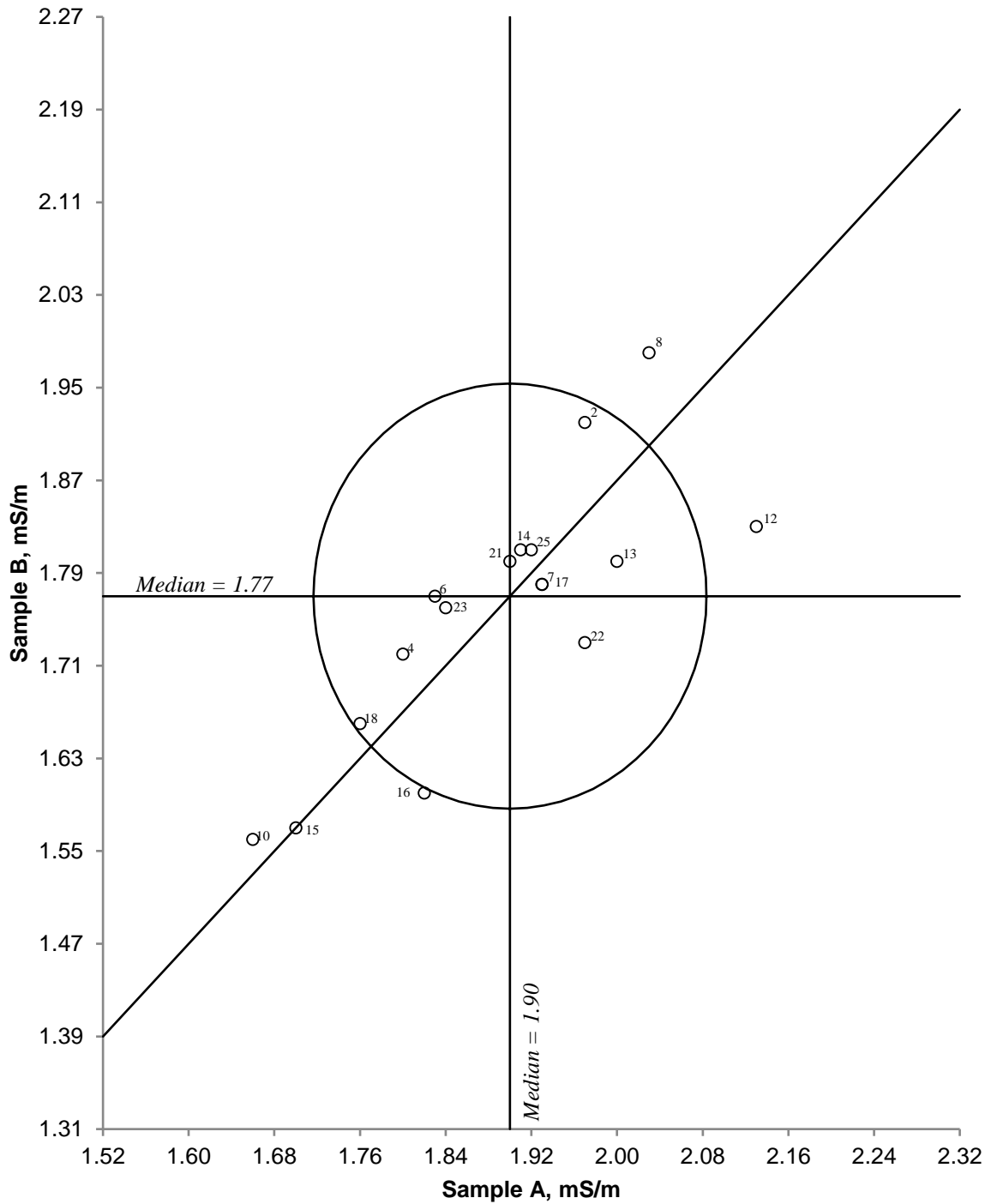
Figure 2. Youden diagram for conductivity. Sample pair AB. Acceptance limit, given by circle, is 10%.

Alkalinity
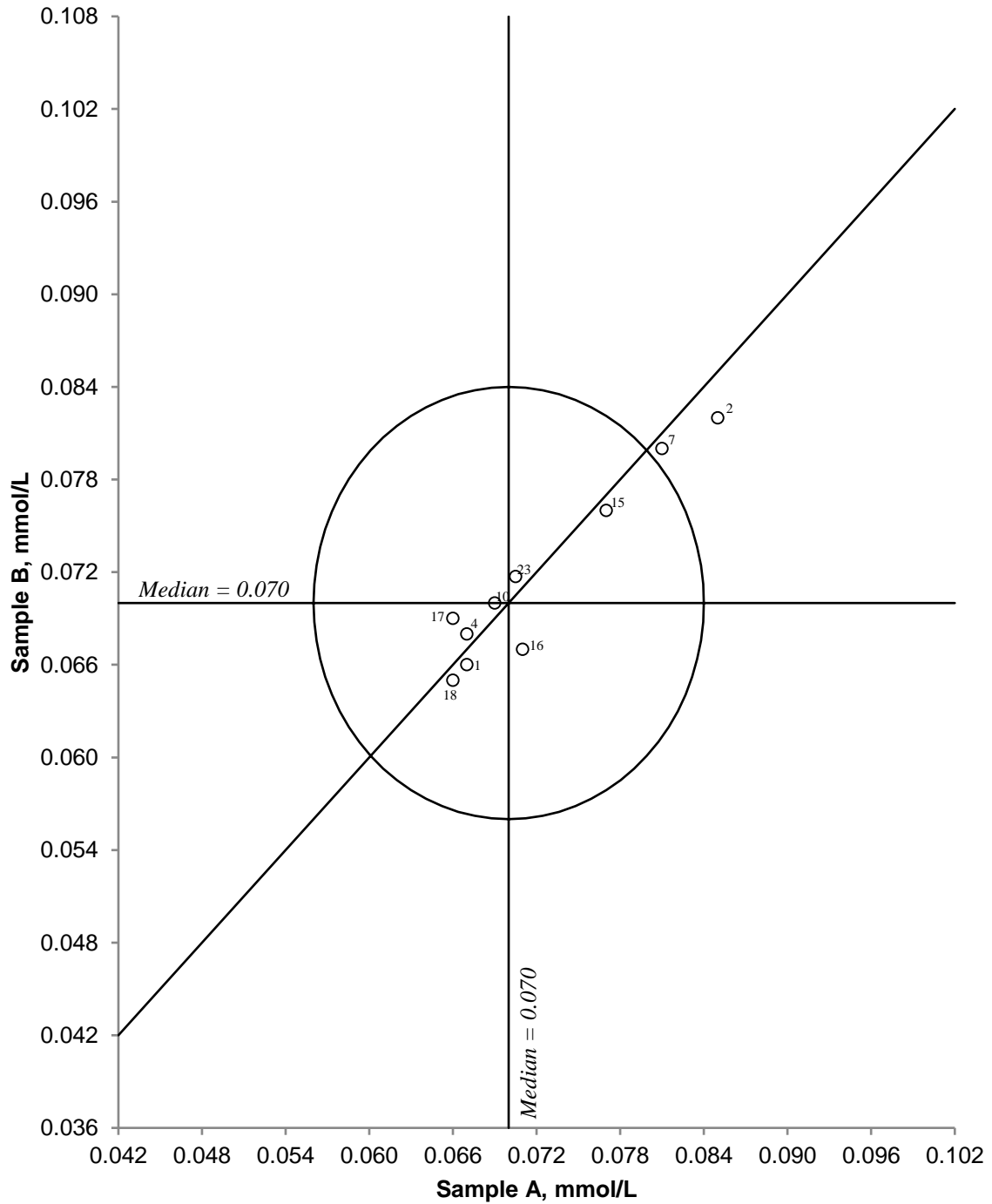
Figure 3. Youden diagram for alkalinity. Sample pair AB. Acceptance limit, given by circle, is 20%.
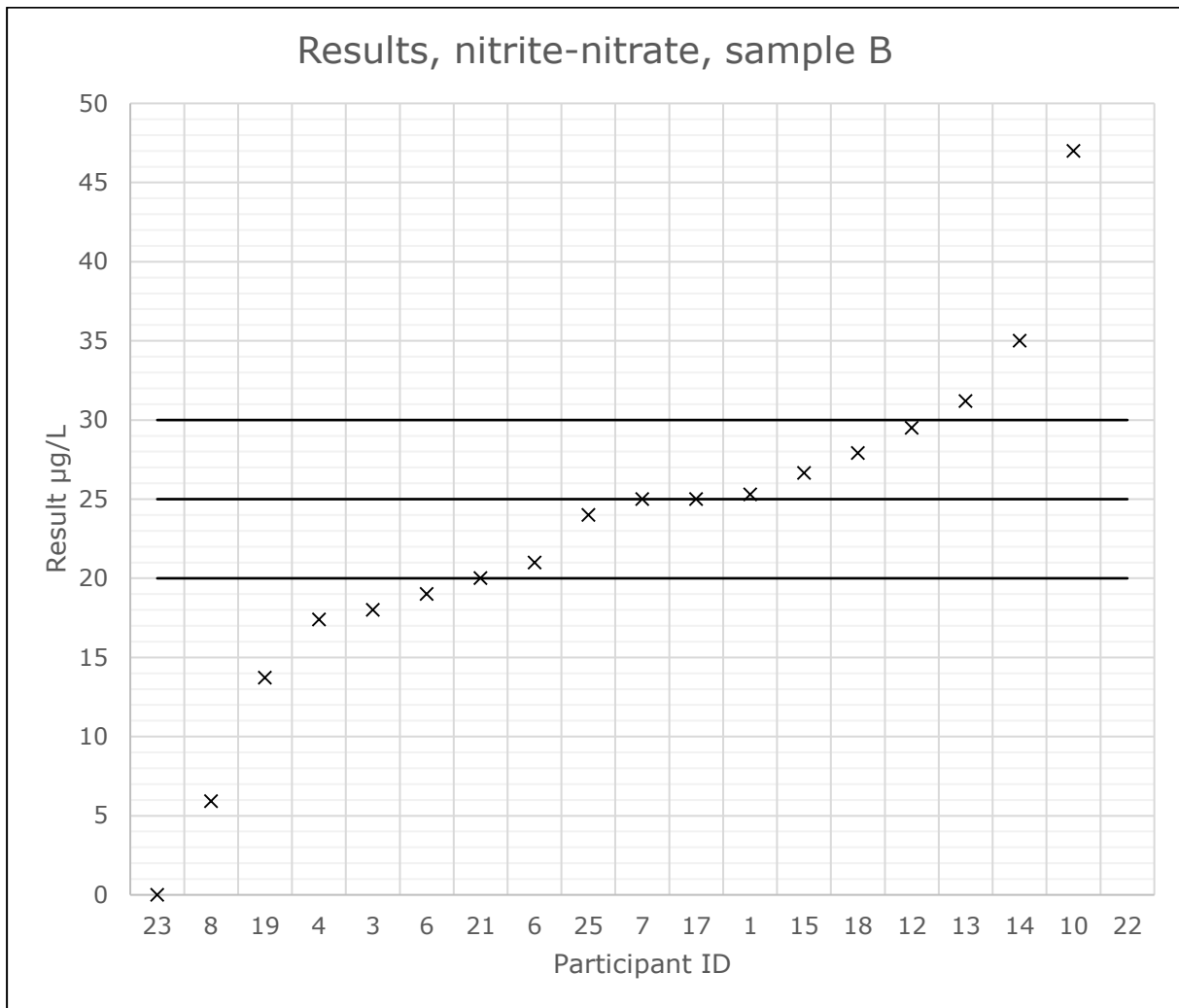
Figure 4. Results within ± 20% of the "true value" for nitrite-nitrate for sample B, calculated separately from sample A.

Chloride
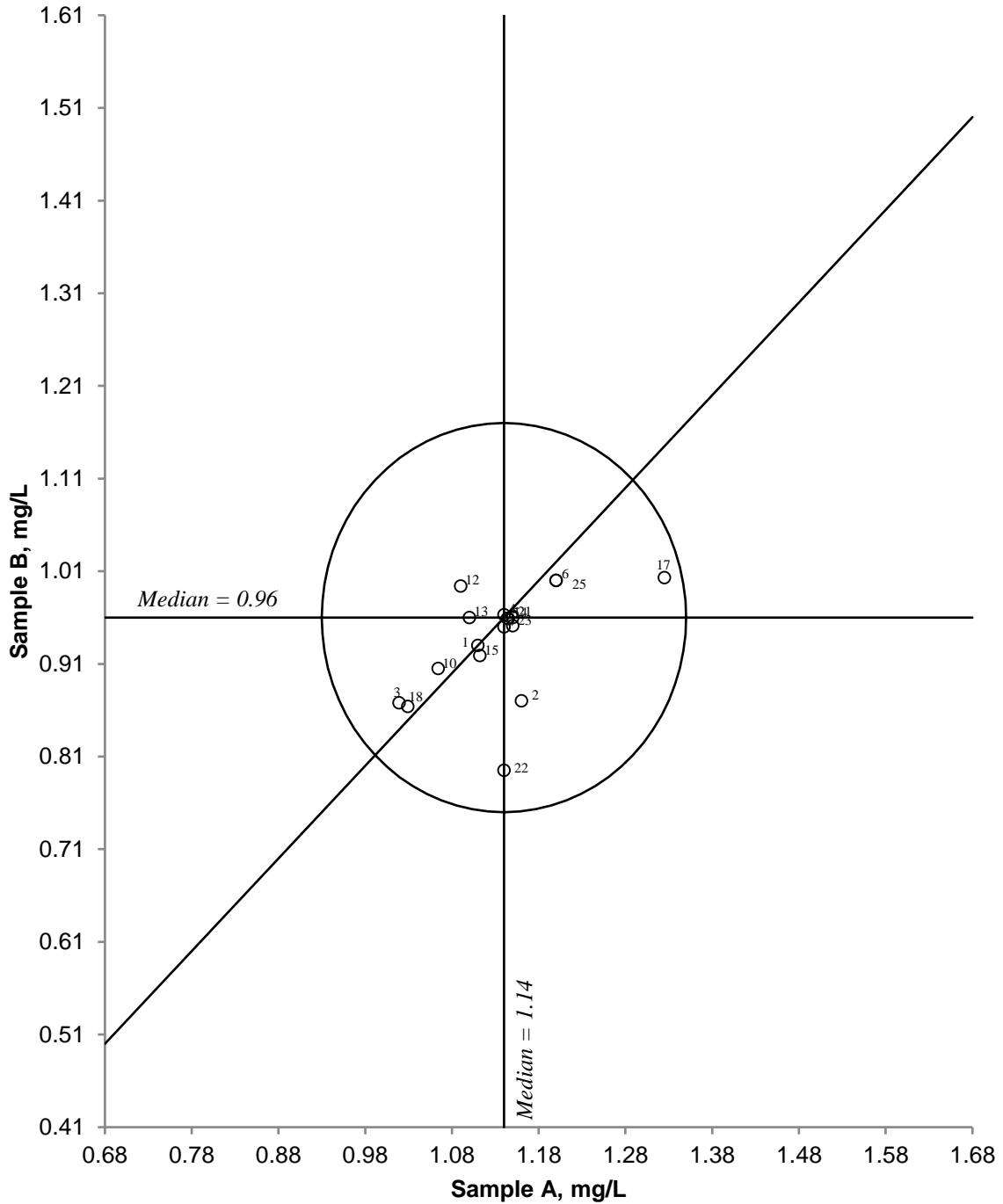
Figure 5. Youden diagram for chloride. Sample pair AB. Acceptance limit, given by circle, is 20%.

Sulphate
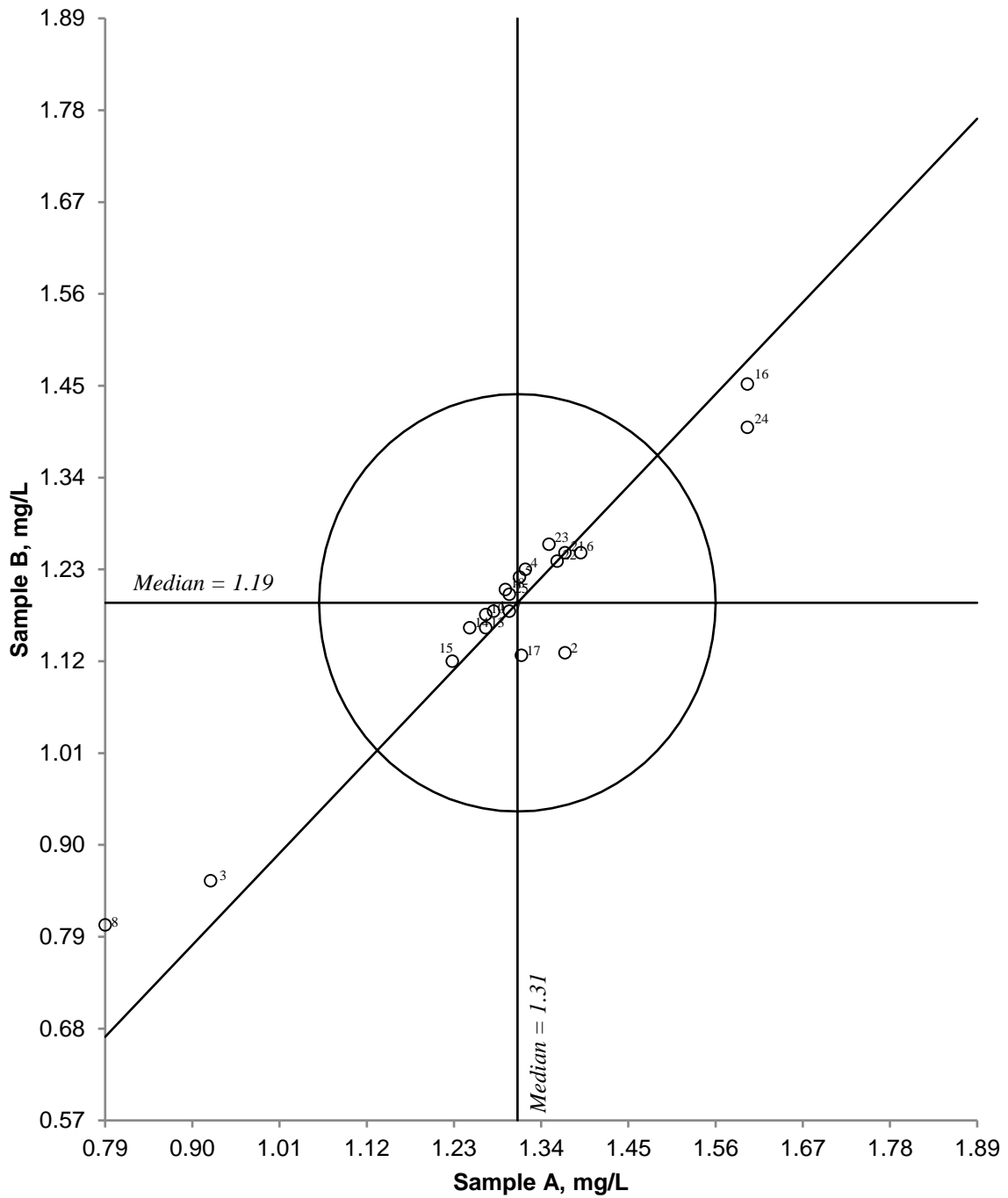
Figure 6. Youden diagram for sulphate. Sample pair AB. Acceptance limit, given by circle, is 20%.

Calcium
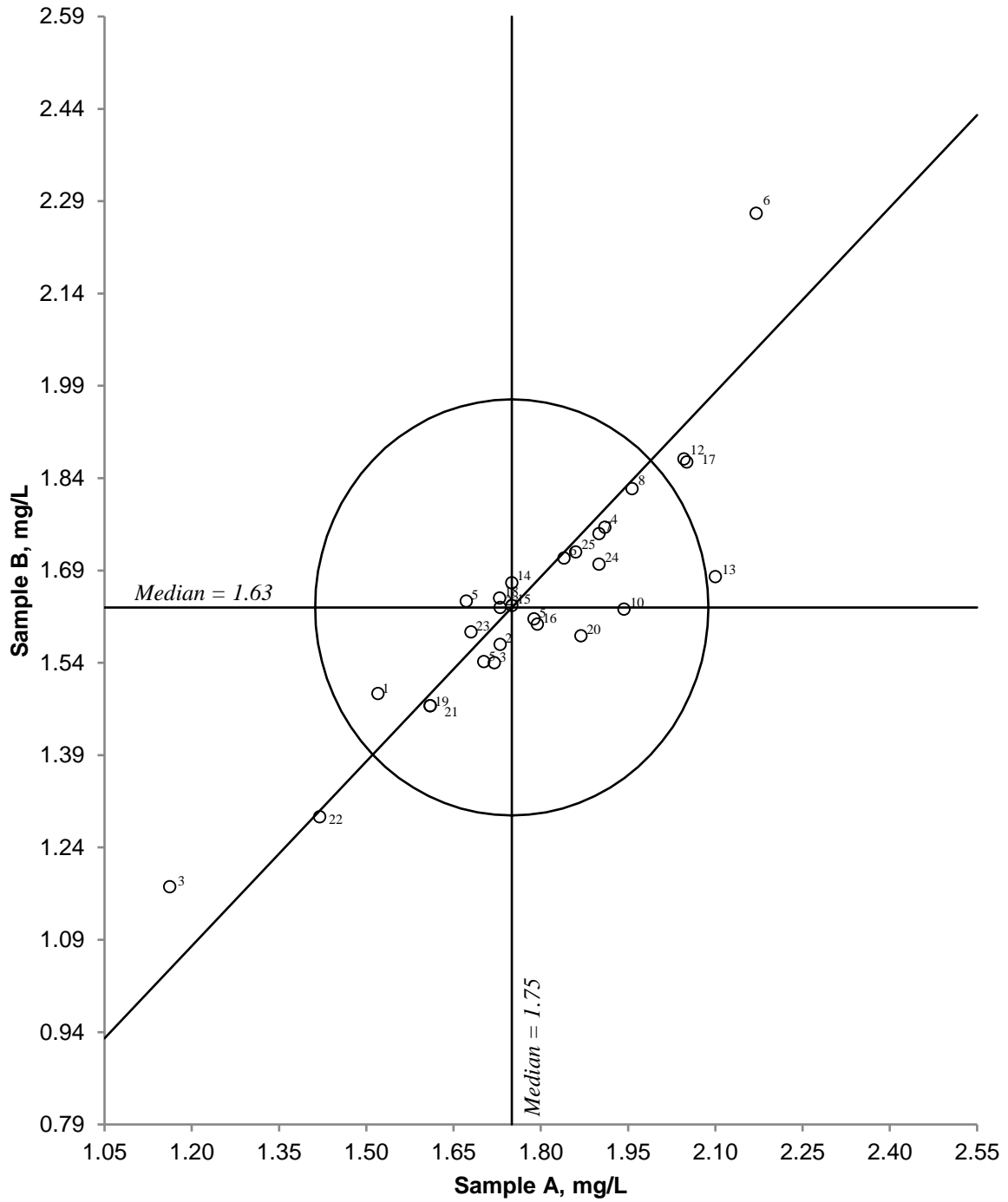
Figure 7. Youden diagram for calcium. Sample pair AB. Acceptance limit, given by circle, is 20%.

Magnesium
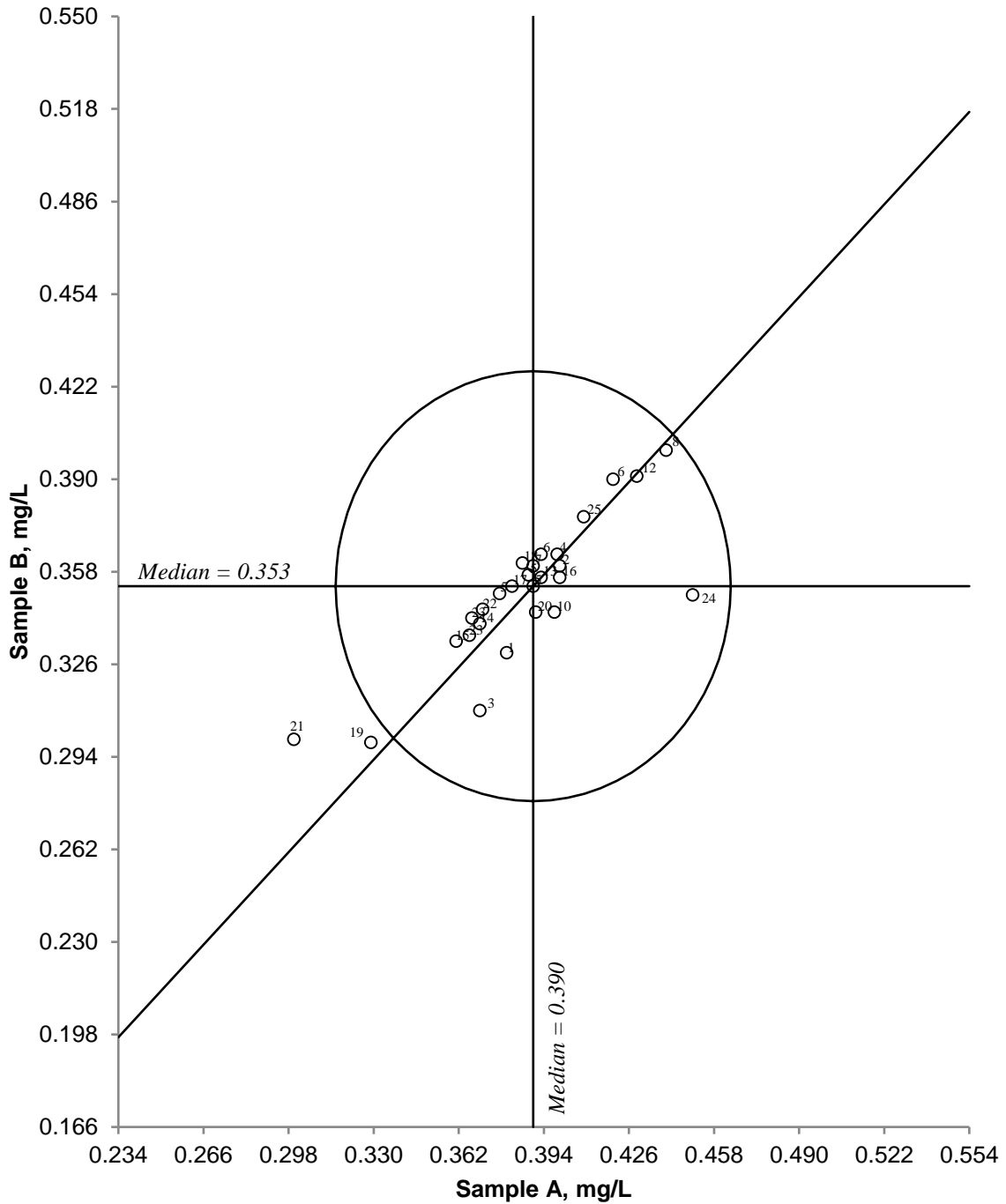
Figure 8. Youden diagram for magnesium. Sample pair AB. Acceptance limit, given by circle, is 20%.

Sodium
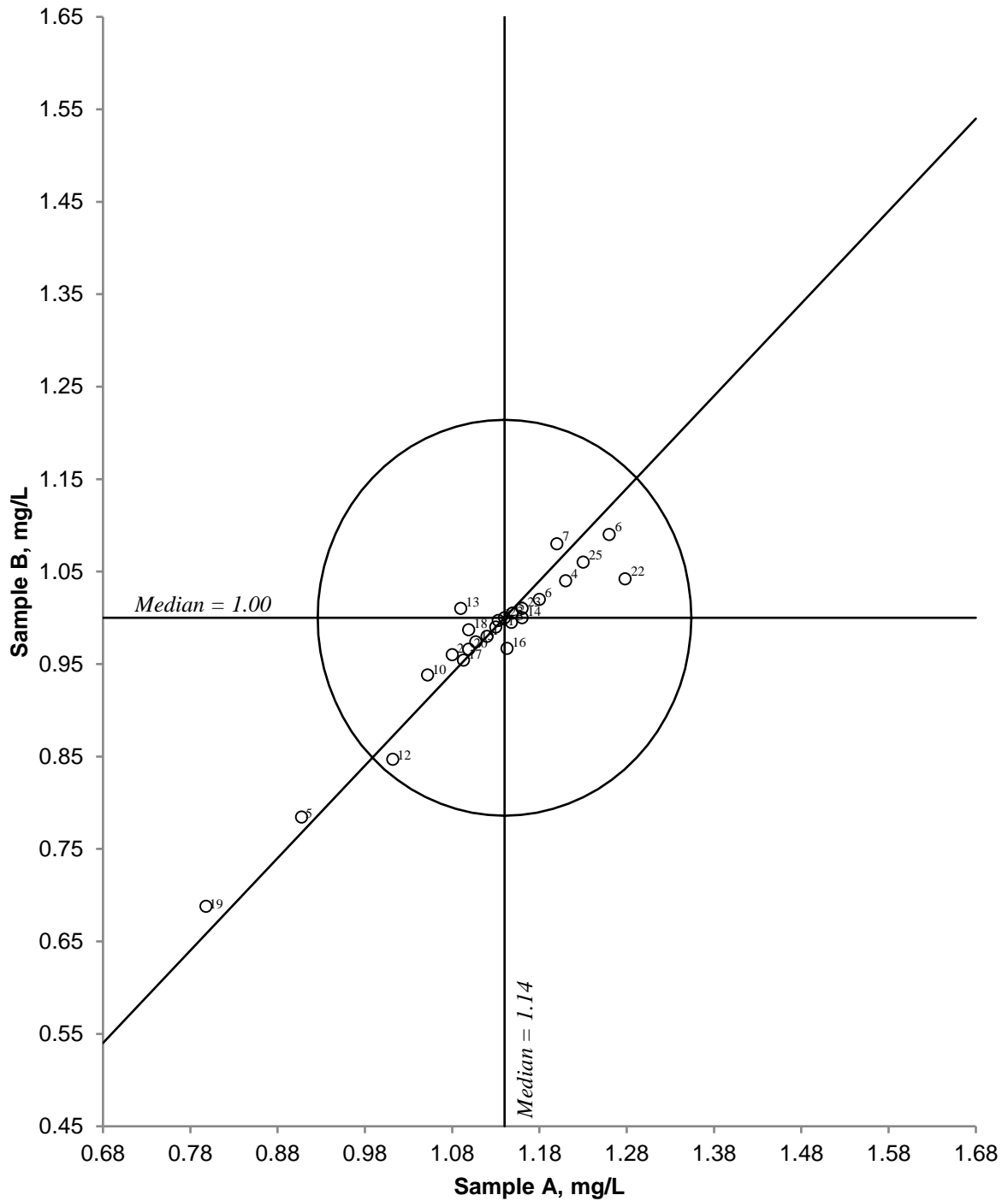
Figure 9. Youden diagram for sodium. Sample pair AB. Acceptance limit, given by circle, is 20%.

Potassium
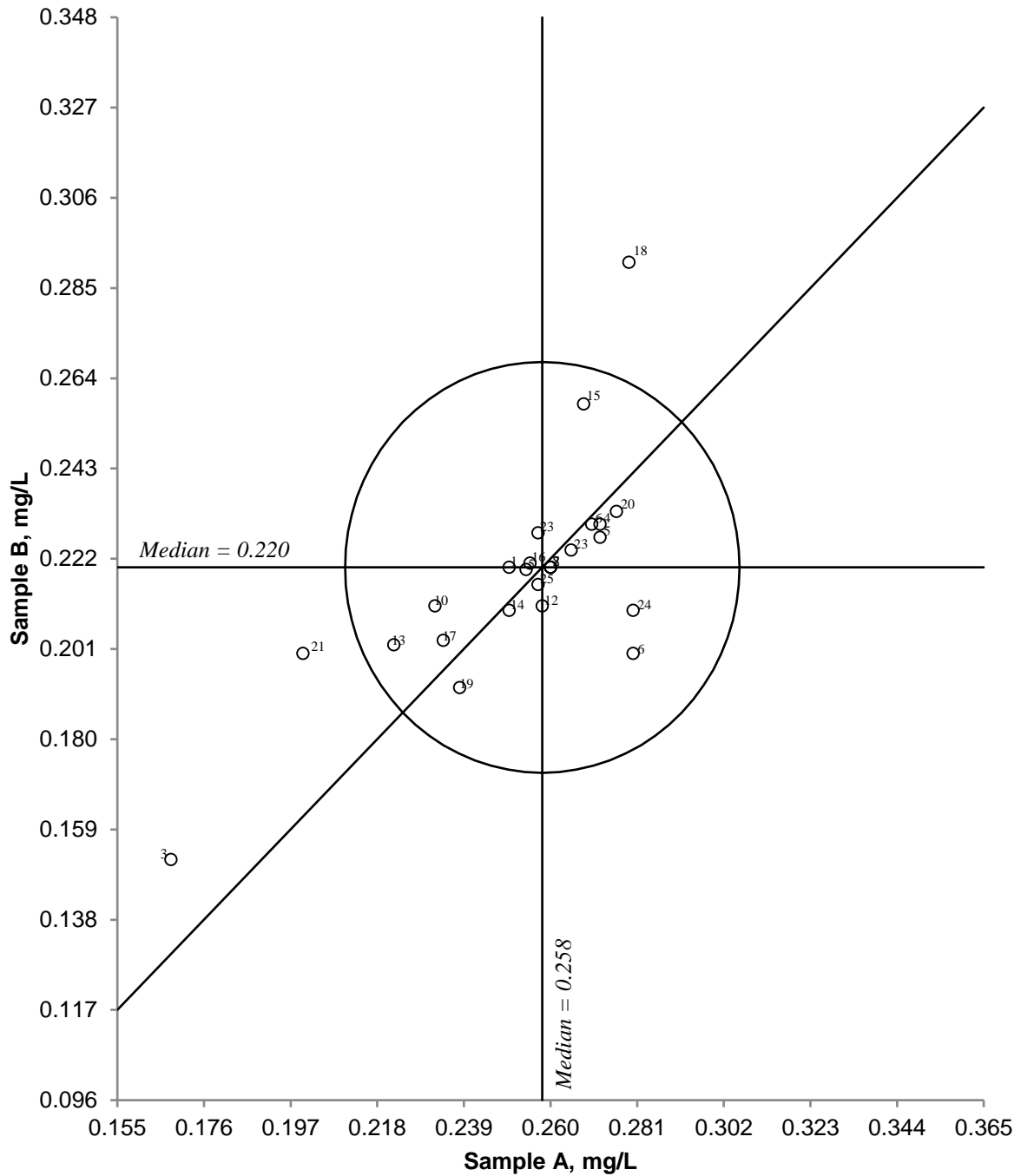
Figure 10. Youden diagram for potassium. Sample pair AB. Acceptance limit, given by circle, is 20%.

Total organic carbon
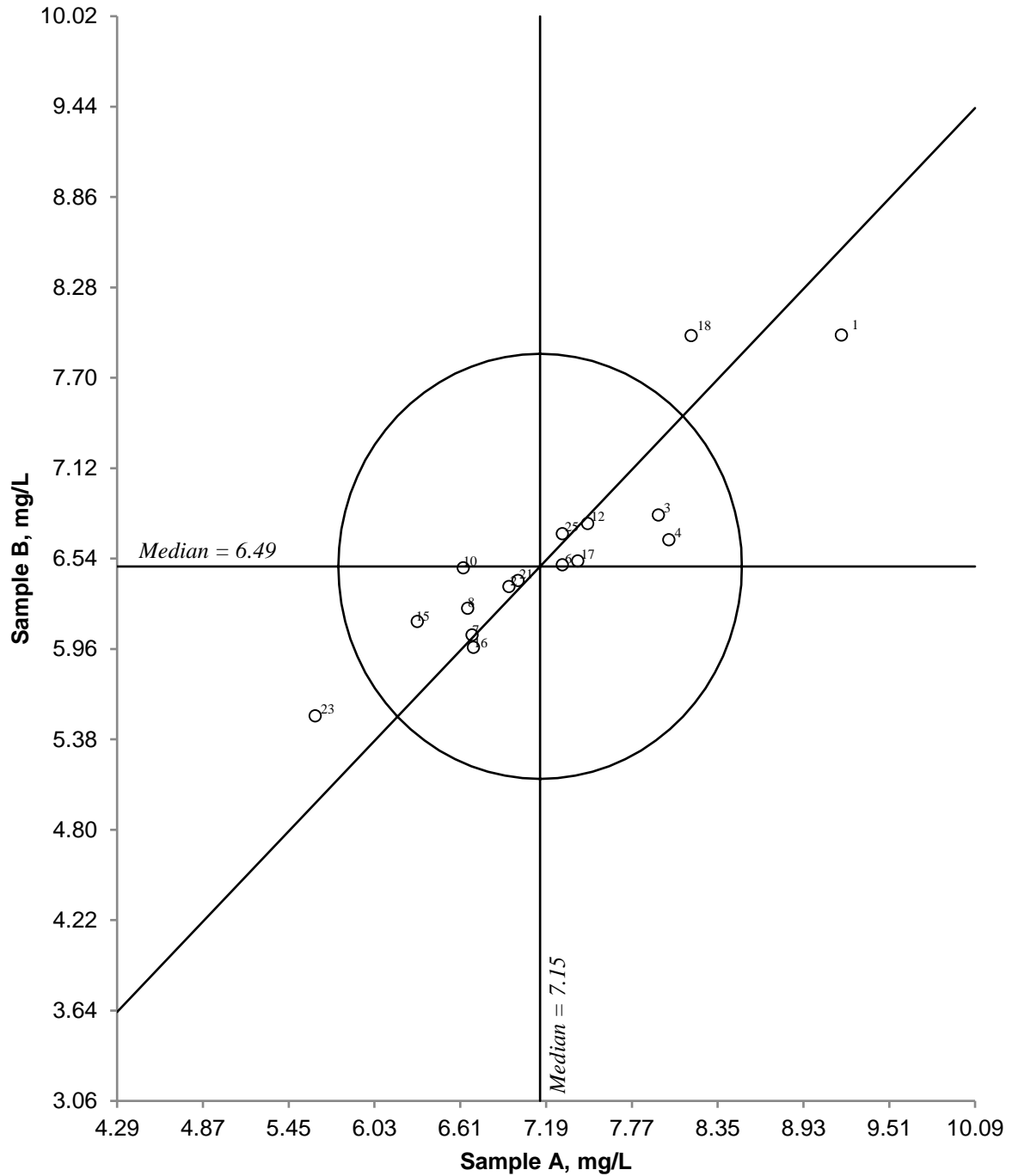
Figure 11. Youden diagram for total organic carbon. Sample pair AB. Acceptance limit, given by circle, is 20%.

Total phosphorous
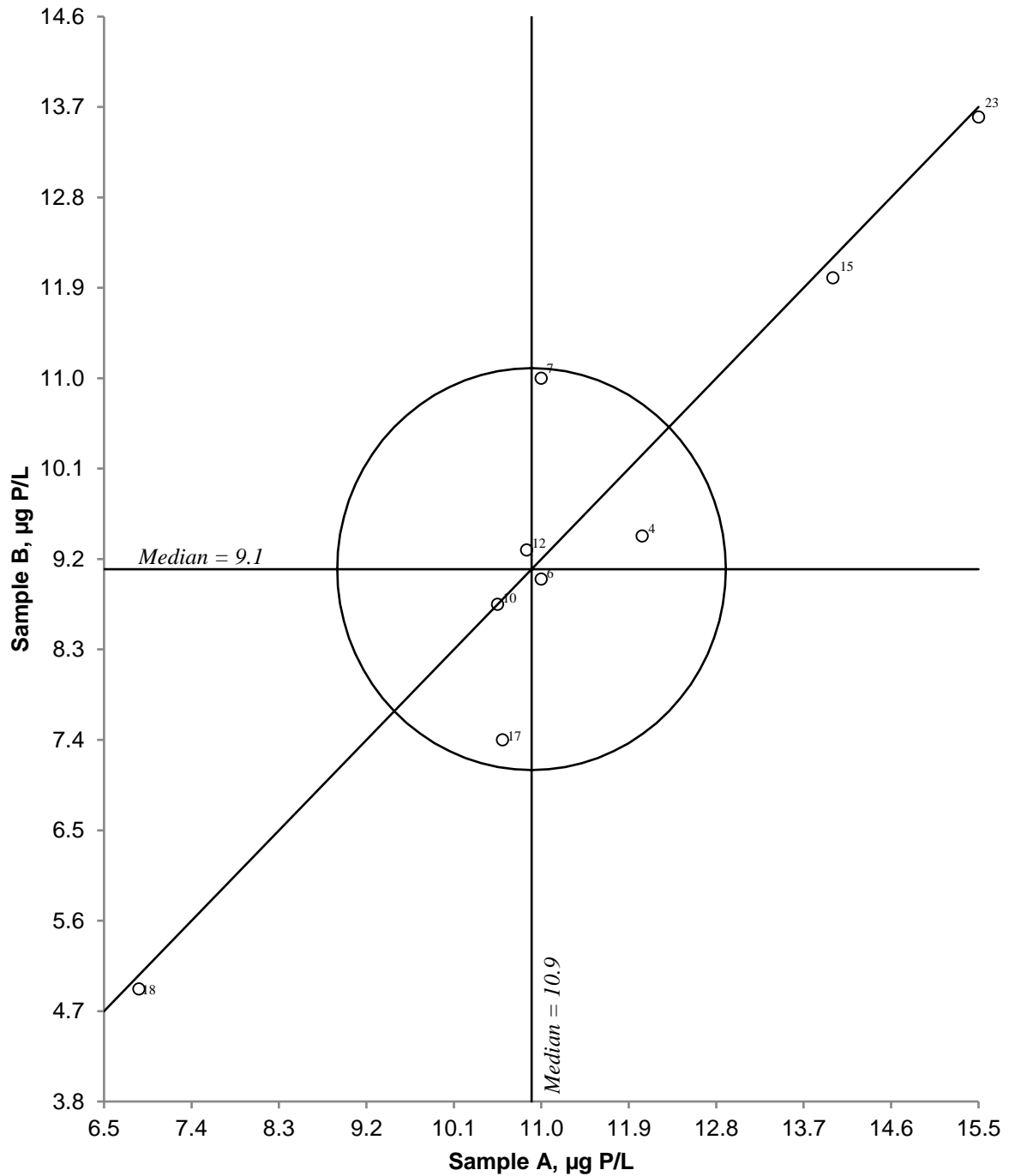
Figure 12. Youden diagram for total phosphorous. Sample pair AB. Acceptance limit, given by circle, is 20%.

Total nitrogen
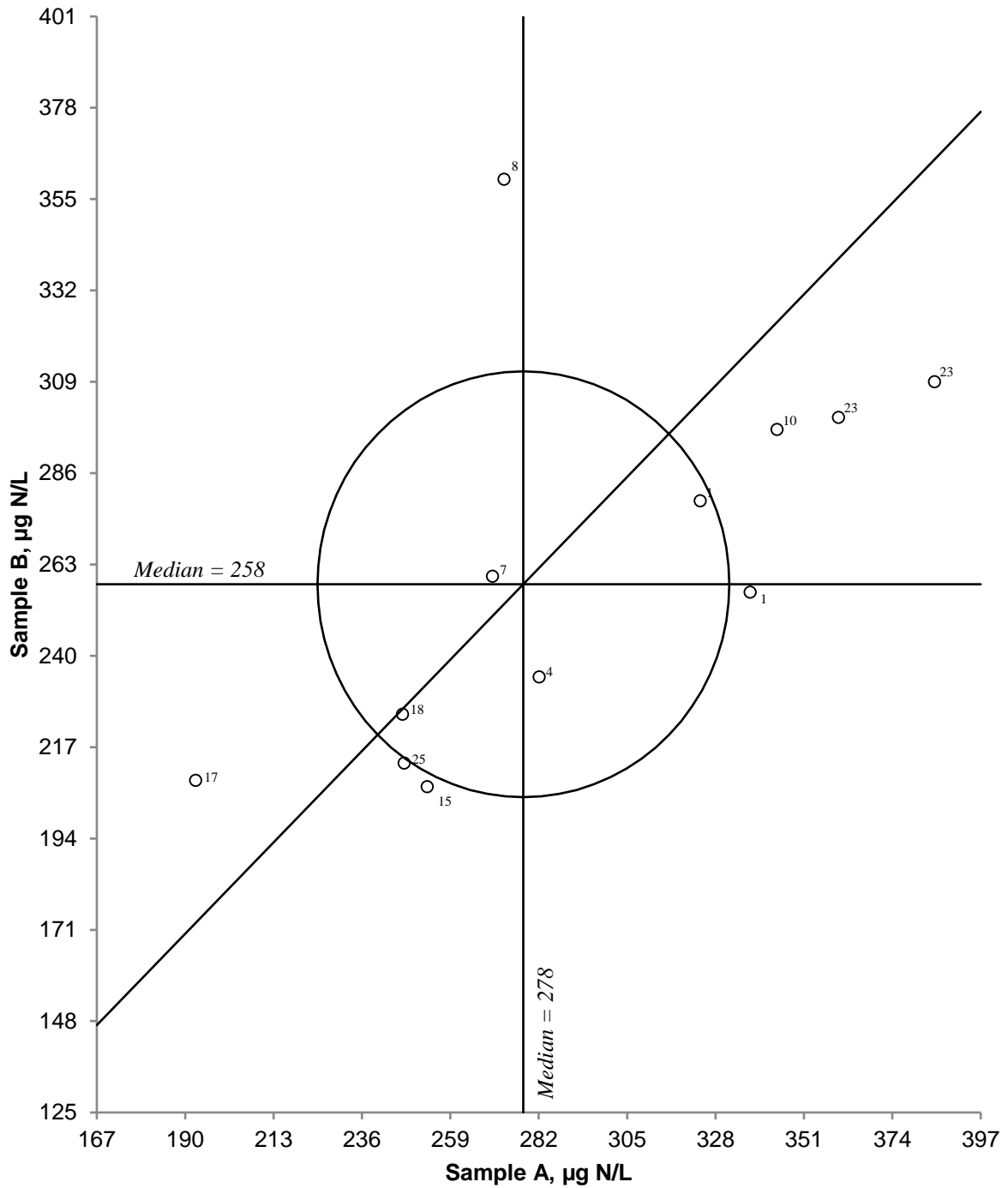
Figure 13. Youden diagram for total nitrogen. Sample pair AB. Acceptance limit, given by circle, is 20%

Aluminium
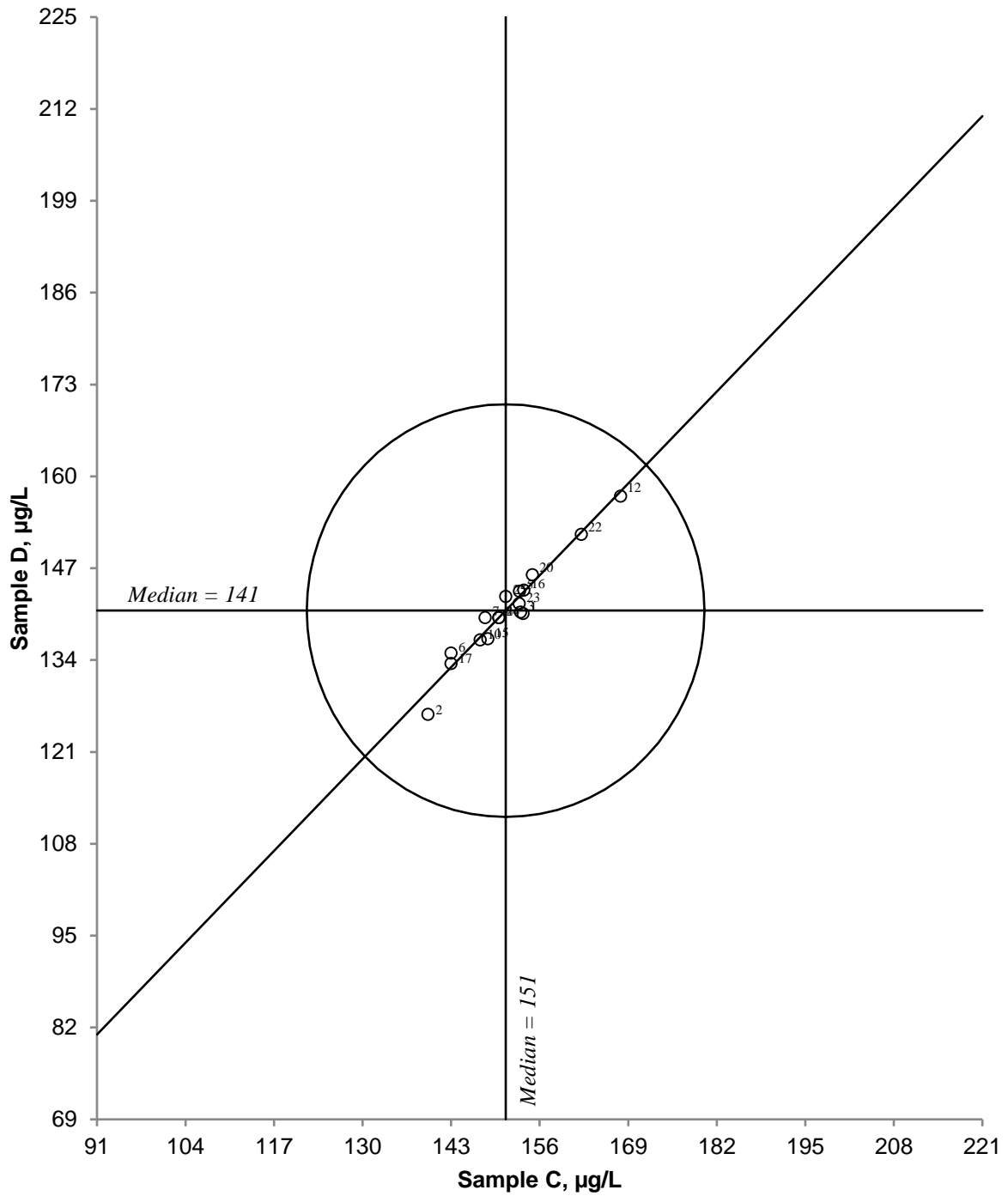
Figure 14. Youden diagram for aluminium. Sample pair CD. Acceptance limit, given by circle, is 20%.

Iron
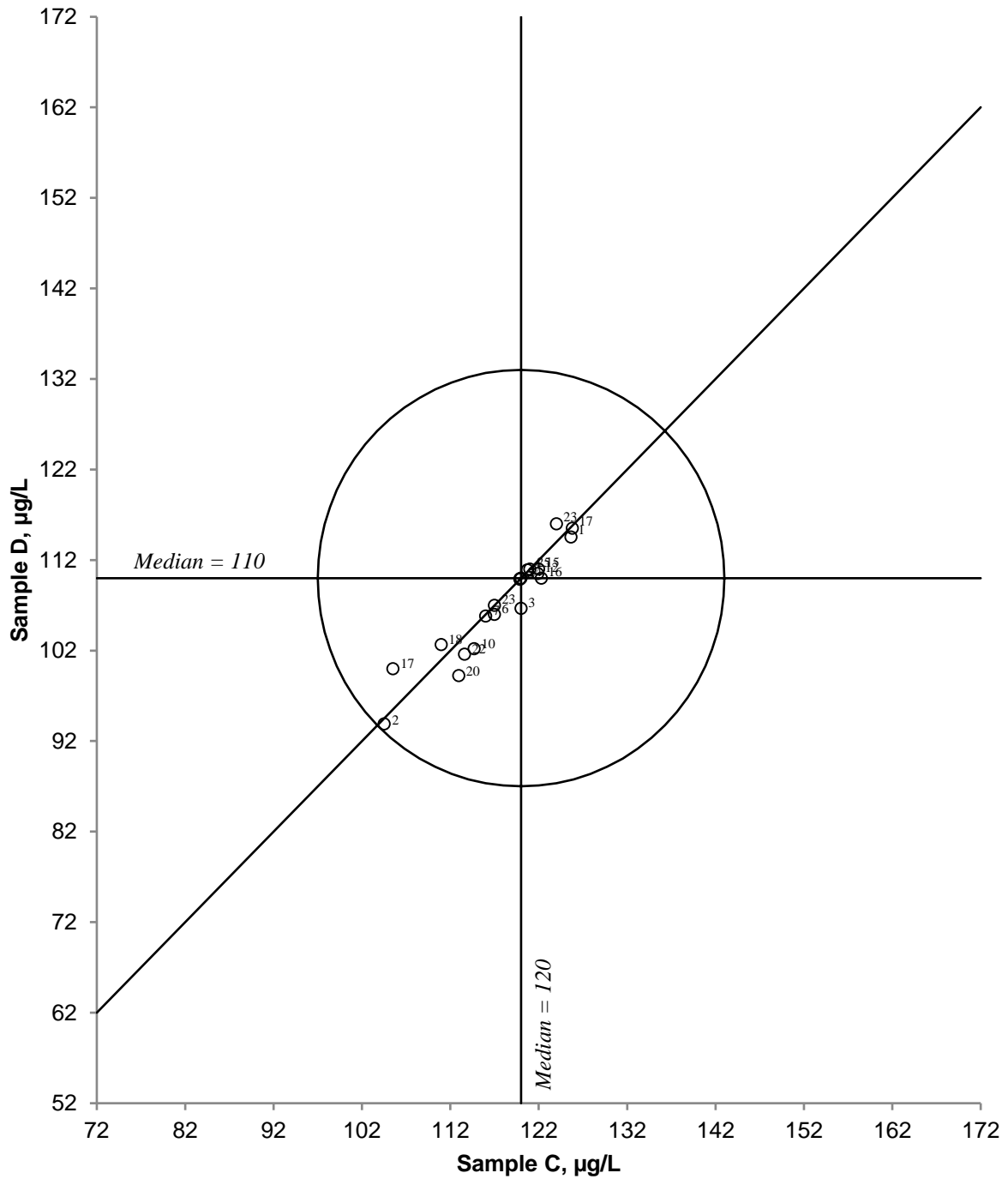
Figure 15. Youden diagram for iron. Sample pair CD. Acceptance limit, given by circle, is 20%.

Manganese
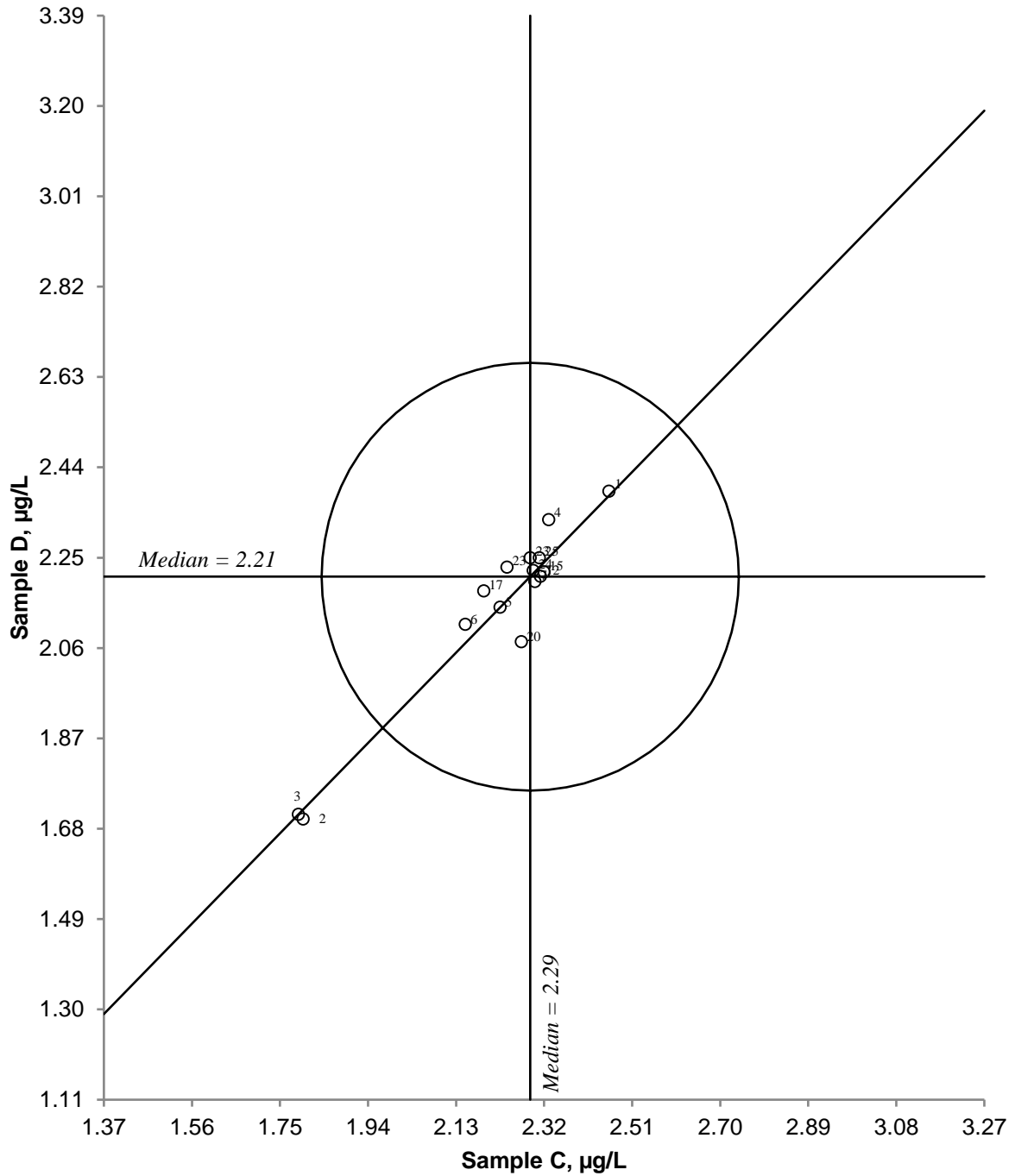
Figure 16. Youden diagram for manganese. Sample pair CD. Acceptance limit, given by circle, is 20%.

Cadmium
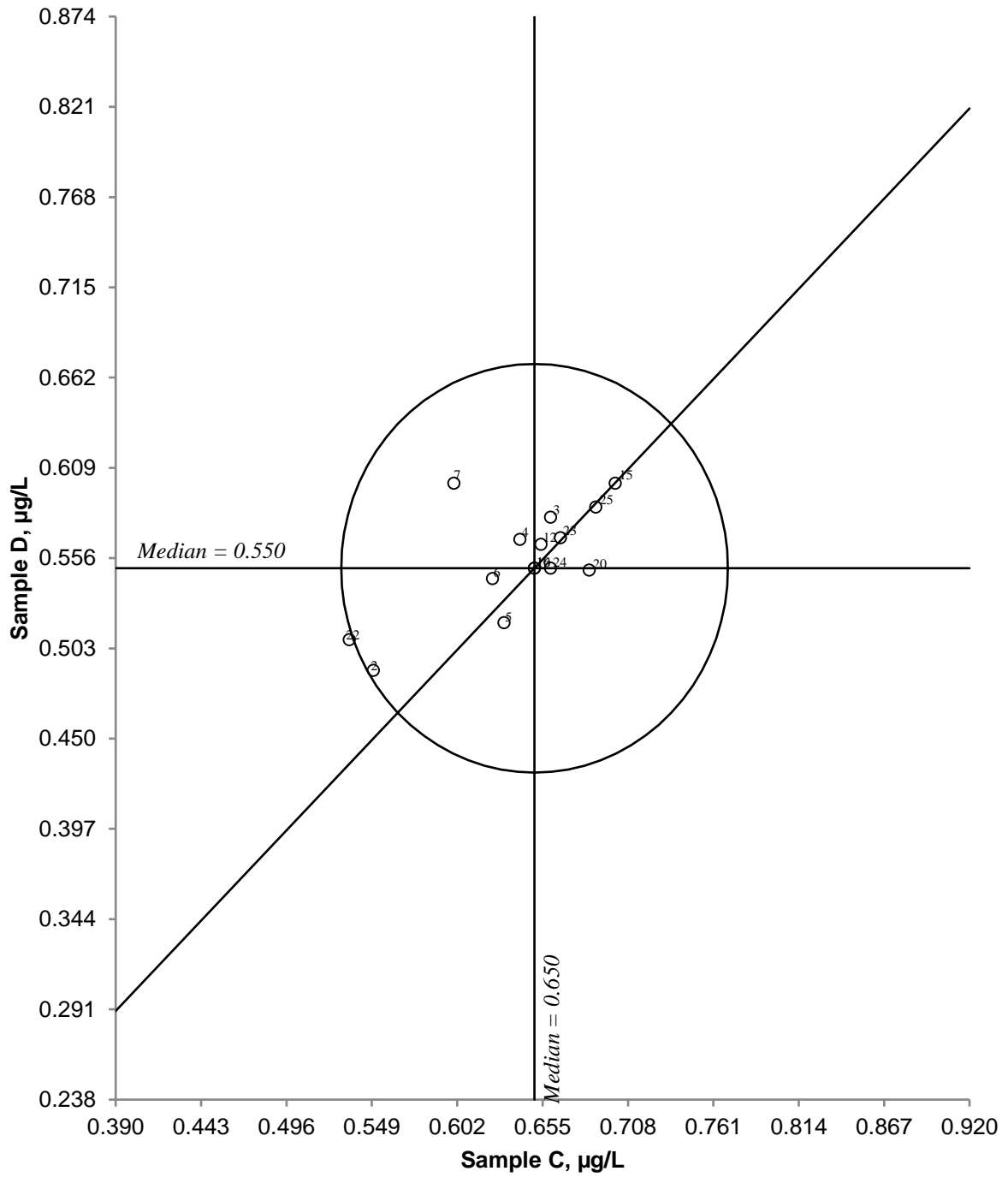
Figure 17. Youden diagram for cadmium. Sample pair CD. Acceptance limit, given by circle, is 20%.

Lead
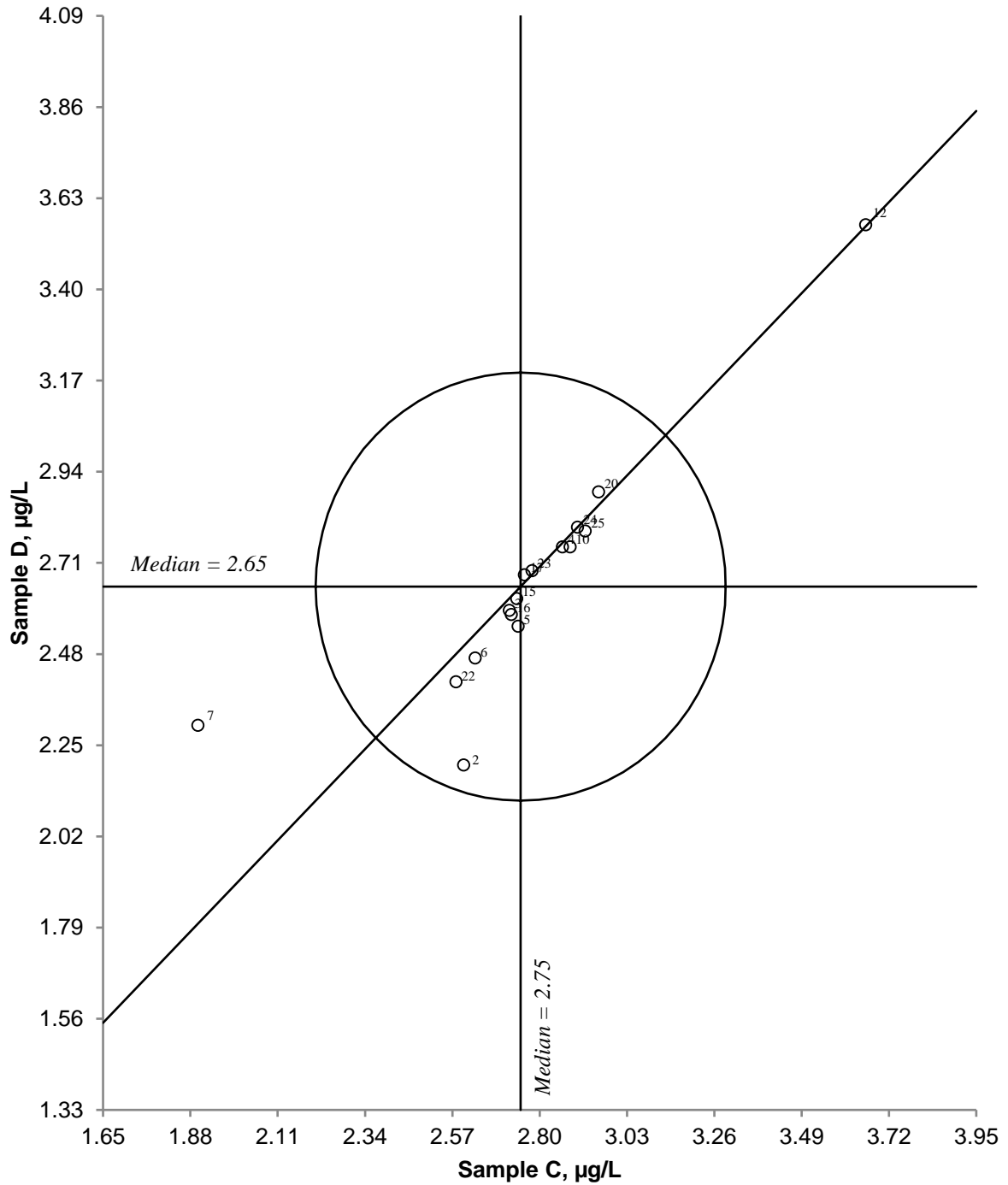
Figure 18. Youden diagram for lead. Sample pair CD. Acceptance limit, given by circle, is 20%.

Copper
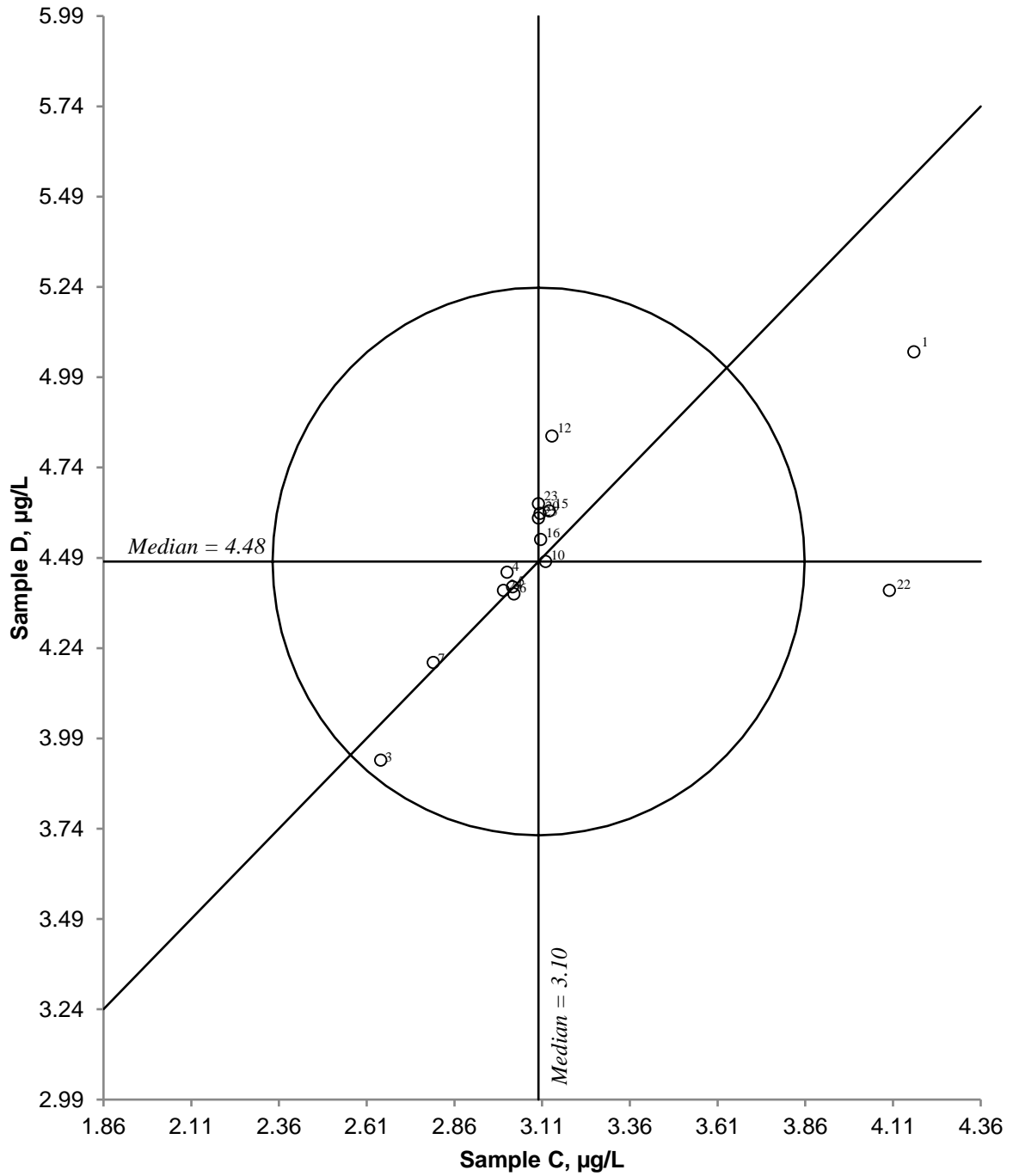
Figure 19. Youden diagram for copper. Sample pair CD. Acceptance limit, given by circle, is 20%.

Nickel
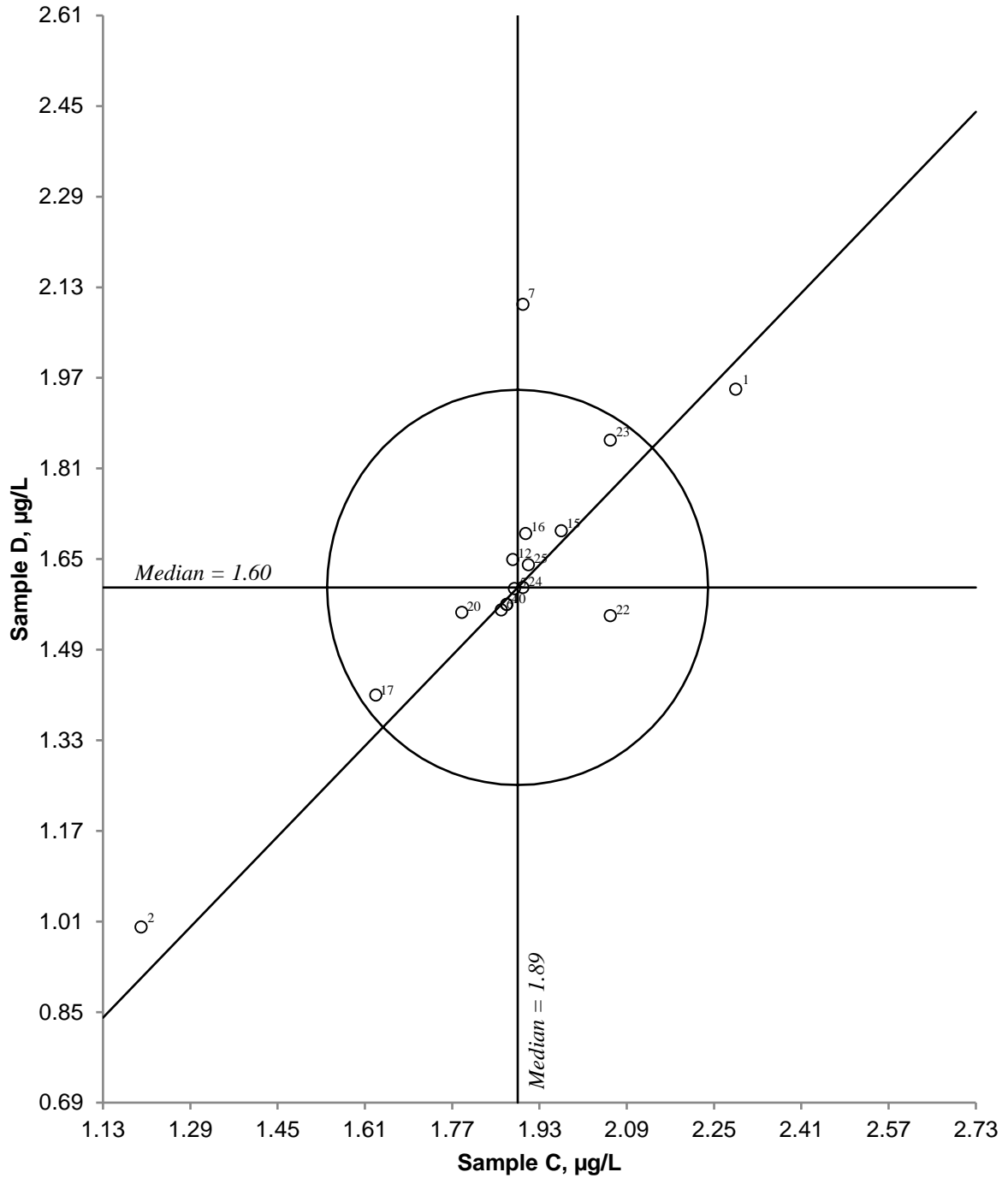
Figure 20. Youden diagram for nickel. Sample pair CD. Acceptance limit, given by circle, is 20%.

Zinc
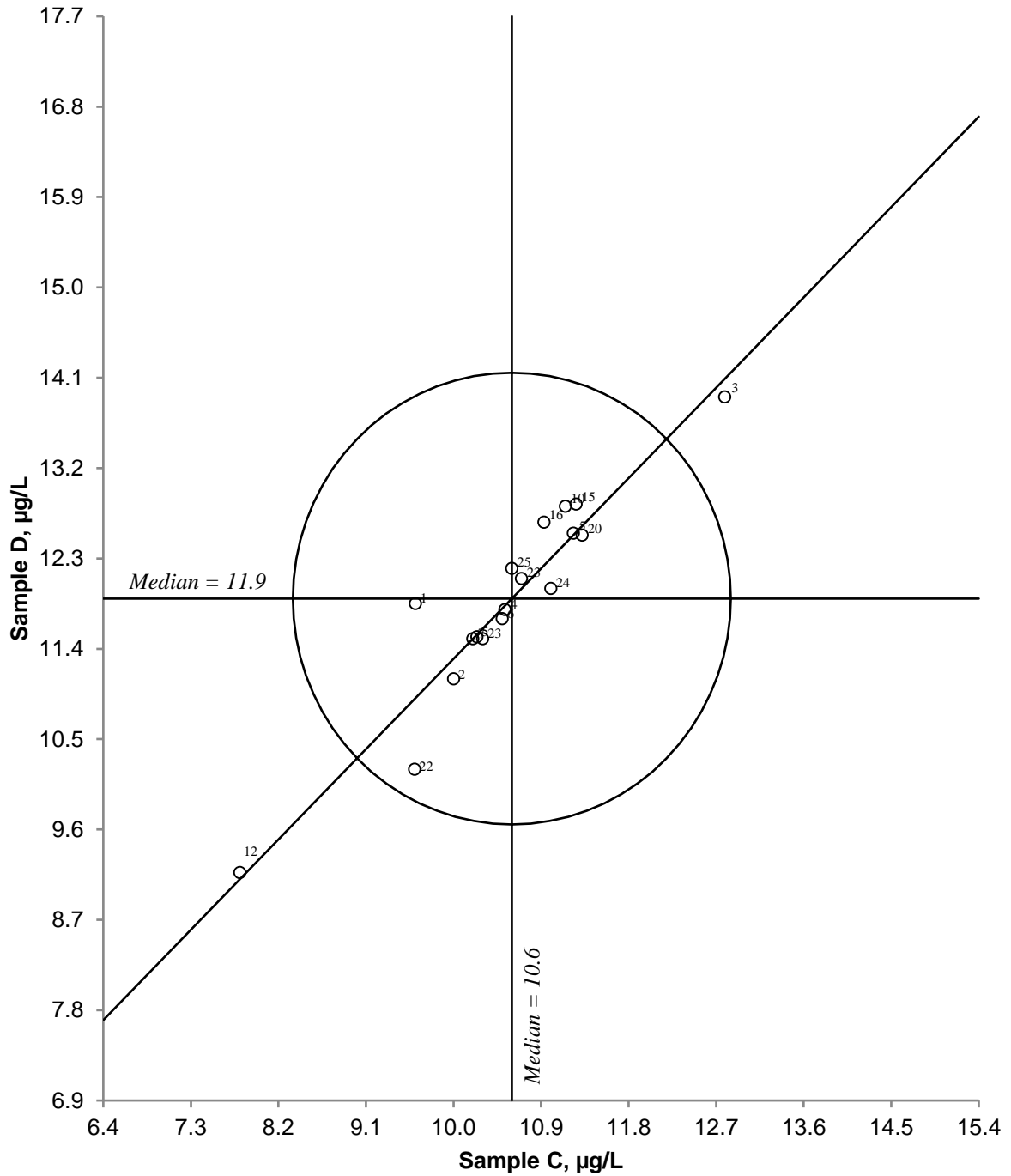
Figure 21. Youden diagram for zinc. Sample pair CD. Acceptance limit, given by circle, is 20%.

4   Literature

1.   ICP Waters Programme Centre 2010. ICP Waters Programme manual. ICP Waters report 105/2010. NIVA SNO 6074-2010. 91p.

2.   Youden, W.J.: Graphical Diagnosis of Interlaboratory Test Results. Industrial Quality Control. 1959, pp 15 - 24.

3.   Youden, W.J., Steiner, E.H.: Statistical Manual of the Association of Official Analytical Chemists. Statistical Techniques for Collaborative Tests. Arlington, 1975.

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 (2022): Statistical methods for use in proficiency testing by interlaboratory comparisons.

Appendix A. The participating laboratories

Table A. 1. Information of the participating laboratories including name, address, and country.

No	Name of Laboratory	Address	Country
1	Centre for Hidrographic Studies	Paseo Bajo Virgen del Puerto, 3, 28005 Madrid	SPAIN
2	Chemical Laboratory, Czech Geological Survey	Geologická 6, 152 00 Prague	CZECH REPUBLIC
3	Servei d'Anàlisi Química i Estructural	STR-UdG Pic de Peguera, 15 17003-Girona	SPAIN
4	Swedish University for Agricultural Sciences Aquatic Sciences and Assesment	Box 7050 750 07 UPPSALA	SWEDEN
5	ISSeP Colfontaine Zoning Schweitzer	Rue de la Platinerie B-7340 COLFONTAINE	BELGIUM
6	Norsk institutt for vannforskning	Økernveien 94 NO-0579 OSLO	NORWAY
7	CNR Institute of Water Research (IRSA)	Largo Tonolli 50 I-28922 VERBANIA Pallanza	ITALY
8	Laboratoire d'écologie fonctionnelle et environnement (EcoLab)	Avenue Agrobiopole 31326 Castanet Tolosan	FRANCE
9	Institute of Environmental Protection-Puszcza Borecka station	Kolektorska 4, 01-692, Warszawa, Poland	POLAND
10	Ufficio del Monitoraggio Ambientale - Laboratorio	Via Mirasole 22 6500 Bellinzona	SWITZERLAND
11	MOEECC, DORSET Laboratory	P.O. Box 39 Dorset, Ontario Canada , P0A 1E0	CANADA
12	Büsgen-Institute - Soil Science of Temperate Ecosystems	D-37077 Goettingen Buesgenweg 2	GERMANY
13	Forest Nutrition and Water Resources Department of Ecology, Technis	H.C.v.Carlowitz-Platz 2 D-85354 Freising Germany	GERMANY
14	Staatliche Betriebsgesellschaft für Umwelt und Landwirtschaft (BfUL)	Haus 5, FB53 Waldheimer Str. 219 D-01683 Nossen	GERMANY
15	Bayerische Landesanstalt fuer Wald und Forstwirtschaft Stabsstelle L.3 - Labor	Hans-Carl-von-Carlowitz-Platz 1 D-85354 Freising	GERMANY
16	Natural Resources Wales Analytical Services (NRWAS)	NRW Analytical Services at Swansea University, Faraday Building 2nd floor, Swansea University Singleton Campus, Swansea, SA2 8PP	UNITED KINGDOM
17	Institut fur Ökologie	Technikerstr. 25 6020 Innsbruck Austria, Europe	AUSTRIA
18	Marine Scotland Science Freshwater Laboratory	Faskally,Pitlochry,Perthshire, PH16 5BB, Scotland.	UNITED KINGDOM

Table A. 1. cont.

No	Name of Laboratory	Address	Country
19	Radbouduniversiteit afd. Ecologie t.a.v. G. Verheggen	Postbus 9010 6500 GL Nijmegen The Netherlands	NETHERLANDS
20	Vlaamse MilieuMaatschappij (VMM) Dienst Laboratorium	Raymonde de Larochelaan 1,9051 Sint-Denijs-Westrem	BELGIUM
21	Staatliche Betriebgesellschaft für Umwelt und Landwirtschaft (BfUL)	Dresdner Straße 183 D-09131 Chemnitz	GERMANY
22	Polish Academy of Sciences Institute of Botany	PAN Instytut Botaniki 31-512 Kraków ul. Lubicz 46	POLAND
23	Estonian Environment Research Centre	Marja 4 D 10617 Tallinn, Estonia	ESTONIA
24	NLS Starcross laboratory Staplake Mount	Starcross labortory, Exeter, EX68FD	UNITED KINGDOM
25	Bayerisches Landesamt fuer Umwelt	Ref 71 Bürgerm-Ulrich-Str. 160 D-86179 Augsburg	GERMANY
26	Lab di Microanalysis DAGRI University of Florence	Via Maragliano, 77 50143 Firenze	ITALY

Table A.2: Overview of the different countries represented by the participating laboratories.

Country	No. of labs.	Country	No. of labs.
Austria	1	Italy	2
Belgium	2	Netherlands	1
Canada	1	Norway	1
Czech Republic	1	Poland	2
Estonia	1	Spain	2
France	1	Sweden	1
Germany	6	Switzerland	1
		United Kingdom	3

Total: 15 countries

Appendix B. Preparation of the samples

Both sample sets, AB and CD, were prepared using water from Nordre Piperen (Northeren Piperen Lake) outside of Oslo, Norway. The lake is a small forest lake, chosen as it has high amounts of humic acid and low amounts of lime.

The water was collected during the 5th of May 2022 and transported to the laboratory using four 25 L plastic containers. The water was allowed to settle for approximately two weeks before filtration through 0.45 µm cellulose acetate membrane filters. The filtered water was collected in four 50 L plastic containers, where it was allowed to settle for four weeks before water was collected for a preliminary analysis. After the results of the preliminary analysis were ready, the below mentioned additions were made to produce sample sets AB and CD.

To produce sample set AB, some amount of phosphorous and nitrogen was added, using Adenosine 5'-monophosphate disodium salt (AMP disodium). This addition was conducted as close as possible to the day of sample shipment to avoid biodegradation. A small amount of $Mg(NO_3)_2 \times 6H_2O$ was also added to both A and B, in order to increase the nitrate concentration, theoretically from around 30 µg/l to around 60 µg/l. This, however, seems to have failed, likely due to instability of the (unpreserved) water over time.

Sample set CD was created by spiking with standard solution of the metals: lead, cadmium, copper, nickel, iron, and zinc. For iron, copper and zinc, the spike was only made in one of the two samples, so that samples C and D would have a proper difference in concentration. Aluminium, and manganese was found to be present in high enough, and differing enough, values without spiking the samples. Sample set CD was conserved by adding nitric acid to a concentration of 0.5% (v/v). The water prepared for sample set AB was distributed to 500 mL bottles and the water for sample set CD to 250 mL bottles. The samples were then shipped a few days later to the participating laboratories.

Appendix C. Statistical treatment of the results

Initial treatment of the analytical results

The results were assessed for the presence of potential outliers which was conducted in two subsequent steps. First, if one or both values in a sample set (AB or CD) was deviating with more than 50% from the true value, that pair of results was omitted. The remaining values were used to calculate the mean and the standard deviation of the distribution. Second, those pairs of results in which one or both values were more than three times the standard deviation higher or lower than the mean value was omitted. The remaining results were used for the final calculation for which the results are presented in Tables D.2.1 – D.2.21. Note that the results omitted from the second step have been marked with the letter "O".

For nitrite-nitrate nitrogen, additional manual calculations were made for sample B, in the same way as mentioned above. The results were then seen on their own, separate from sample A. These manually calculated results are written in parenthesis in Table D.2.4. The results omitted in this manual calculation have been marked with "O*"

Estimation the "true value" and uncertainty

For each variable, the "true value" is the median of the reported results after excluding strongly deviating values (i.e. outliers). Thus, the true value is the consensus value from the participants and the corresponding uncertainty is based on the method given in ISO 13528 (2022), Annex C (algorithm A).

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 $|x_i - m|$ (i = 1, 2 …. p)

New value for the robust standard deviation is then calculated according to equations (C.7) – (C.10) 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 7.7 in ISO 13528 (2022):

$$\mu_X = 1.25 \; x \; \frac{S^*}{\sqrt{p}}$$

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

U= 2 $\times u_X$

It is important to note that there are some limitations to this approach for estimating 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.

The Youden statistical test

The measurement results reported to the intercomparison test was assessed using the method of Youden. This procedure requires that two samples are analyzed for each parameter (e.g. A and B) and that each laboratory reports only one result for each sample and analytical variable. The results for sample A and B are plotted in a coordinate system in which the "true value" of sample A constitutes the x-axis and the "true value" of sample B the y-axis. Then, by plotting the individual results from each laboratory in the chart, producing one point for each laboratory (result from sample A along the x-axis and result from sample B along the y-axis), the distribution of the results among the laboratories is visualized (see Figures 1 - 21). Patterns in the distribution of the results can reveal systematic and/or random errors among the participating laboratories.

For example, if the results are affected by random errors only, the points will be spread randomly around the origo of the Youden chart. However, if systematic effects are influencing the results (e.g. from the use of different deviating analytical methods), the points in the chart will be distributed in a characteristic elliptical pattern along a 45° line in the chart. 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 is indicated in the Youden chart by a circle around the origo. The distance from the center of the circle and the point of an individual laboratory is a measure of the absolute error of the result. 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. Thus, the location of the point of each laboratory in the Youden's diagram provides important information of the size and type of analytical error (random or systematic) present in the dataset, making it possible to indicate what is the source of deviation from the consensus of the participating laboratories.

Table C.1. Uncertainty of the calculated "true value" for each parameter.

Parameter and unit	Sample	True value	Total no.	Robust std.dev.	Uncertainty	Expanded uncertainty
pH	A	6.17	22	0.206	0.055	0.110
Units	B	6.21	22	0.188	0.050	0.100
Conductivity	A	1.90	19	0.154	0.044	0.089
mS/m	B	1.77	19	0.141	0.040	0.081
Alkalinity	A	0.070	10	0.0070	0.0028	0.0055
mmol/L	B	0.070	10	0.0066	0.0026	0.0052

Table C.1. cont.

Parameter and unit	Sample	True value	Total no.	Robust std.dev.	Uncertainty	Expanded uncertainty
Nitrate+nitrite-nitrogen, µg N/L	B*	25.0	15	9.63	2.76	5.52
Chloride	A	1.14	18	0.055	0.016	0.032
mg/L	B	0.96	18	0.056	0.016	0.033
Sulphate	A	1.31	20	0.075	0.021	0.042
mg/L	B	1.19	20	0.075	0.021	0.042
Calcium	A	1.75	28	0.189	0.045	0.089
mg/L	B	1.63	27	0.129	0.031	0.062
Magnesium	A	0.390	27	0.0233	0.0056	0.0112
mg/L	B	0.353	27	0.0197	0.0047	0.0095
Sodium	A	1.14	25	0.074	0.018	0.037
mg/L	B	1.00	24	0.045	0.012	0.023
Potassium	A	0.258	25	0.0187	0.0047	0.0093
mg/L	B	0.220	25	0.0147	0.0037	0.0074
Total organic carbon	A	7.15	16	0.775	0.242	0.485
mg/L	B	6.49	16	0.435	0.136	0.272
Total phosphorous	A	10.9	11	3.52	1.33	2.66
µg P/L	B	9.1	8	2.26	1.00	1.99
Total nitrogen	A	278	12	63.4	22.9	45.7
µg N/L	B	258	12	49.3	17.8	35.6
Aluminium	C	151	17	5.9	1.8	3.6
µg/L	D	141	17	5.5	1.7	3.3
Iron	C	120	21	5.5	1.5	3.0
µg/L	D	110	21	5.9	1.6	3.2
Manganese	C	2.29	15	0.094	0.030	0.061
µg/L	D	2.21	15	0.114	0.037	0.074
Cadmium	C	0.650	15	0.0396	0.0128	0.0256
µg/L	D	0.550	15	0.0336	0.0108	0.0217
Lead	C	2.75	16	0.161	0.050	0.100
µg/L	D	2.65	16	0.220	0.069	0.138
Copper	C	3.10	15	0.115	0.037	0.074
µg/L	D	4.48	15	0.216	0.070	0.139
Nickel	C	1.89	17	0.161	0.049	0.097
µg/L	D	1.60	17	0.193	0.058	0.117
Zinc	C	10.6	19	0.81	0.23	0.46
µg/L	D	11.9	18	0.81	0.24	0.48

Appendix D. Results reported by the participating laboratories

Table D.1. Results reported by the participating laboratories.

Lab. nr.	pH		Conductivity, mS/m		Alkalinity, mmol/L		Nitrate + nitrite-nitrogen, µg N/L		Chloride, mg/L		Sulphate, mg/L		Calcium, mg/L	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B
1	6.08	6.16	1.51	1.40	0.067	0.066	13.1	25.3	1.11	0.93	1.28	1.18	1.52	1.49
2	6.46	6.38	1.97	1.92	0.085	0.082			1.16	0.87	1.37	1.13	1.73	1.57
3	5.53	5.76	1.48	1.54			5.0	18.0	1.02	0.87	0.92	0.86	1.72	1.54
4	6.01	6.06	1.80	1.72	0.067	0.068	0.8	17.4	1.14	0.96	1.32	1.23	1.91	1.76
5	6.33	6.33	18.97	18.06					1.14	0.96	1.31	1.22	1.79	1.61
6	6.41	6.47	1.83	1.77	0.112	0.114	-2.0	19.0	1.20	1.00	1.39	1.25	2.17	2.27
7	6.08	6.07	1.93	1.78	0.081	0.080	5.0	25.0	1.14	0.95	1.30	1.18	1.90	1.75
8	6.18	6.16	2.03	1.98	0.120	0.113	0.2	5.9	0.43	0.44	0.79	0.80	1.96	1.82
10	6.09	6.13	1.66	1.56	0.069	0.070	41.0	47.0	1.06	0.91	1.27	1.18	1.94	1.63
12	6.16	6.26	2.13	1.83			63.5	29.5	1.09	0.99	0.44	0.40	2.05	1.87
13	6.45	6.48	2.00	1.80			12.7	31.2	1.10	0.96	1.27	1.16	2.10	1.68
14	6.10	6.16	1.91	1.81			20.0	35.0	1.15	0.96	1.25	1.16	1.75	1.67
15	6.67	6.65	1.70	1.57	0.077	0.076	13.6	26.7	1.11	0.92	1.23	1.12	1.75	1.63
16	6.32	6.33	1.82	1.60	0.071	0.067					1.60	1.45	1.79	1.60
17	6.43	6.44	1.93	1.78	0.066	0.069	0.0	25.0	1.33	1.00	1.32	1.13	2.05	1.87
18	6.18	6.21	1.76	1.66	0.066	0.065	11.4	27.9	1.03	0.86	1.30	1.21	1.73	1.65
19	6.09	6.11			0.130	0.130	0.0	13.7	0.36	0.16			1.61	1.47
20													1.87	1.58
21	6.50	6.50	1.90	1.80			0.0	20.0	1.15	0.96	1.37	1.25	1.61	1.47
22	6.37	6.32	1.97	1.73			51.5	94.2	1.14	0.80	1.36	1.24	1.42	1.29
23	6.14	6.21	1.84	1.76	0.070	0.072	0.0	0.0	1.15	0.95	1.35	1.26	1.73	1.63
24											1.60	1.40	1.90	1.70
25	6.02	6.03	1.92	1.81			0.0	24.0	1.20	1.00	1.30	1.20	1.86	1.72

Table D.1. cont.

Lab. nr.	Magnesium, mg/L		Sodium, mg/L		Potassium, mg/L		Total organic carbon, mg/L		Total phosphorous, µg P/L		Total nitrogen, µg N/L		Aluminium, µg/L	
	A	B	A	B	A	B	A	B	A	B	A	B	C	D
1	0.380	0.330	1.12	0.98	0.250	0.220	9.19	7.97	26.8	8.4	337	256	154	141
2	0.400	0.360	1.08	0.96	0.260	0.220	6.94	6.36					140	126
3	0.370	0.310	2.70	1.52	0.290	0.000	7.95	6.82					153	141
4	0.399	0.364	1.21	1.04	0.272	0.230	8.02	6.66	12.0	9.4	282	235	150	140
5	0.390	0.353	1.15	1.01	0.272	0.227			0.0	3.7			153	144
6	0.420	0.390	1.26	1.09	0.280	0.200	7.30	6.50	11.0	9.0			143	135
7	0.390	0.360	1.20	1.08	0.260	0.220	6.69	6.05	11.0	11.0	270	260	148	140
8	0.440	0.400	1.13	1.00	0.260	0.220	6.66	6.22	24.3	26.5	273	360		
10	0.398	0.344	1.05	0.94	0.232	0.211	6.63	6.48	10.6	8.8	344	297	147	137
12	0.429	0.391	1.01	0.85	0.258	0.211	7.47	6.77	10.9	9.3			168	157
13	0.393	0.356	1.09	1.01	0.222	0.202			6.0	6.0				
14	0.370	0.340	1.16	1.00	0.250	0.210								
15	0.361	0.334	1.11	0.97	0.268	0.258	6.32	6.14	14.0	12.0	253	207	148	137
16	0.400	0.356	1.14	0.97	0.255	0.221	6.70	5.97					154	144
17	0.382	0.353	1.09	0.95	0.234	0.203	7.40	6.53	10.6	7.4	193	209	143	134
18	0.386	0.361	1.10	0.99	0.279	0.291	8.17	7.97	6.9	4.9	247	225		
19	0.329	0.299	0.80	0.69	0.238	0.192			0.0	0.0				
20	0.391	0.344	1.10	0.97	0.276	0.233			33.0	26.0			155	146
21	0.300	0.300	1.13	0.99	0.200	0.200	7.00	6.40	16.0	15.0				
22	0.371	0.345	1.28	1.04	0.525	0.302							162	152
23	0.367	0.342	1.16	1.01	0.265	0.224	5.63	5.53	15.5	13.6	385	309	153	142
24	0.450	0.350	1.70	1.00	0.280	0.210							150	140
25	0.409	0.377	1.23	1.06	0.257	0.216	7.30	6.70	17.0	14.0	247	213	151	143

Table D.1. cont.

Lab. nr.	Iron, µg/L		Manganese, µg/L		Cadmium, µg/L		Lead, µg/L		Copper, µg/L		Nickel, µg/L		Zinc, µg/L	
	C	D	C	D	C	D	C	D	C	D	C	D	C	D
1	126	115	2.46	2.39			5.01	3.20	4.17	5.06	2.29	1.95	9.6	11.9
2	105	94	1.80	1.70	0.550	0.490	2.60	2.20			1.20	1.00	10.0	11.1
3	120	107	1.79	1.71	0.660	0.580	2.72	2.59	2.65	3.93	1.02	0.72	12.8	13.9
4	121	111	2.33	2.33	0.641	0.567	2.86	2.75	3.01	4.45	1.87	1.57	10.5	11.8
5	120	110	2.23	2.15	0.631	0.518	2.74	2.55	3.03	4.41	1.88	1.60	11.2	12.6
6	117	106	2.15	2.11	0.624	0.544	2.63	2.47	3.03	4.39	1.86	1.56	10.5	11.7
7	116	106	2.30	2.20	0.600	0.600	1.90	2.30	2.80	4.20	1.90	2.10	10.2	11.5
8														
10	115	102			0.650	0.550	2.88	2.75	3.12	4.48	1.87	1.57	11.2	12.8
12	122	111	2.31	2.21	0.654	0.564	3.66	3.56	3.14	4.83	1.88	1.65	7.8	9.2
13														
14														
15	122	111	2.32	2.22	0.700	0.600	2.74	2.62	3.13	4.62	1.97	1.70	11.3	12.8
16	122	110			0.650	0.550	2.73	2.58	3.11	4.54	1.91	1.70	10.9	12.7
17	126	116	2.19	2.18	0.120	0.110	2.76	2.68			1.63	1.41	8.3	6.9
18	111	103												
19														
20	113	99	2.27	2.07	0.684	0.549	2.96	2.89	3.10	4.61	1.79	1.56	11.3	12.5
21														
22	114	102	3.20	3.40	0.535	0.508	2.58	2.41	4.10	4.40	2.06	1.55	9.6	10.2
23	124	116	2.29	2.25	0.666	0.568	2.78	2.69	3.10	4.64	2.06	1.86	10.7	12.1
24	120	110	2.30	2.22	0.660	0.550	2.90	2.80	3.00	4.40	1.90	1.60	11.0	12.0
25	121	111	2.31	2.25	0.688	0.586	2.92	2.79	3.10	4.60	1.91	1.64	10.6	12.2

Table D.2.1. Statistics - pH

Sample A

Analytical method: All
Unit: Units

Number of participants	22	Range	1.14
Number of omitted results	0	Variance	0.06
True value	6.17	Standard deviation	0.24
Mean value	6.22	Relative standard deviation	3.8%
Median value	6.17	Relative error	0.7%

Analytical results in ascending order:

3	5.53	23	6.14	6	6.41
4	6.01	5	6.15	17	6.43
25	6.02	12	6.16	13	6.45
1	6.08	18	6.18	2	6.46
7	6.08	8	6.18	21	6.50
10	6.09	16	6.32	15	6.67
19	6.09	5	6.33		
14	6.10	22	6.37		

Sample B

Analytical method: All
Unit: Units

Number of participants	22	Range	0.89
Number of omitted results	0	Variance	0.04
True value	6.21	Standard deviation	0.20
Mean value	6.25	Relative standard deviation	3.2%
Median value	6.21	Relative error	0.6%

Analytical results in ascending order:

3	5.76	14	6.16	2	6.38
25	6.03	5	6.18	17	6.44
4	6.06	18	6.21	6	6.47
7	6.07	23	6.21	13	6.48
19	6.11	12	6.26	21	6.50
10	6.13	22	6.32	15	6.65
8	6.16	5	6.33		
1	6.16	16	6.33		

O = Omitted result

Table D.2.2. Statistics - Conductivity

Sample A

Analytical method: All
Unit: mS/m

Number of participants	21	Range	0.65	
Number of omitted results	2	Variance	0.03	
True value	1.90	Standard deviation	0.17	
Mean value	1.85	Relative standard deviation	9.1%	
Median value	1.90	Relative error	-2.8%	

Analytical results in ascending order:

3	1.48	6	1.83	22	1.97	
1	1.51	23	1.84	2	1.97	
10	1.66	21	1.90	13	2.00	
15	1.70	14	1.91	8	2.03	
18	1.76	25	1.92	12	2.13	
4	1.80	7	1.93	5	18.77	O
16	1.82	17	1.93	5	18.97	O

Sample B

Analytical method: All
Unit: mS/m

Number of participants	21	Range	0.58	
Number of omitted results	2	Variance	0.02	
True value	1.77	Standard deviation	0.14	
Mean value	1.73	Relative standard deviation	8.2%	
Median value	1.77	Relative error	-2.4%	

Analytical results in ascending order:

1	1.40	22	1.73	25	1.81	
3	1.54	23	1.76	14	1.81	
10	1.56	6	1.77	12	1.83	
15	1.57	7	1.78	2	1.92	
16	1.60	17	1.78	8	1.98	
18	1.66	21	1.80	5	17.81	O
4	1.72	13	1.80	5	18.06	O

O = Omitted result

Table D.2.3. Statistics - Alkalinity

Sample A

Analytical method: All
Unit: mmol/L

Number of participants	14	Range	0.019
Number of omitted results	4	Variance	0.000
True value	0.070	Standard deviation	0.007
Mean value	0.072	Relative standard deviation	9.4%
Median value	0.070	Relative error	2.8%

Analytical results in ascending order:

1	0.034	O	10	0.069	2	0.085	
18	0.066		23	0.070	6	0.112	O
17	0.066		16	0.071	8	0.120	O
1	0.067		15	0.077	19	0.130	O
4	0.067		7	0.081			

Sample B

Analytical method: All
Unit: mmol/L

Number of participants	14	Range	0.017
Number of omitted results	4	Variance	0.000
True value	0.070	Standard deviation	0.006
Mean value	0.071	Relative standard deviation	8.3%
Median value	0.070	Relative error	2.1%

Analytical results in ascending order:

1	0.035	O	17	0.069	2	0.082	
18	0.065		10	0.070	8	0.113	O
1	0.066		23	0.072	6	0.114	O
16	0.067		15	0.076	19	0.130	O
4	0.068		7	0.080			

O = Omitted result

Table D.2.4. Statistics - Nitrate + nitrite-nitrogen

Sample A

Analytical method: All
Unit: µg N/L

Number of participants	19	Range	2.2
Number of omitted results	15	Variance	0.9
True value	12.9	Standard deviation	0.9
Mean value	12.7	Relative standard deviation	7.4%
Median value	12.9	Relative error	-1.7%

Analytical results in ascending order:

6	-2.0	O	8	0.2	O	15	13.6	
6	-1.0	O	4	0.8	O	14	20.0	O
19	0.0	O	7	5.0	O	10	41.0	O
25	0.0	O	3	5.0	O	22	51.5	O
17	0.0	O	18	11.4		12	63.5	O
23	0.0	O	13	12.7				
21	0.0	O	1	13.1				

Sample B   Results calculated from sample B only.

Analytical method: All
Unit: µg N/L

Number of participants	19	Range	21.3
Number of omitted results	4	Variance	33.2
True value	25.0	Standard deviation	5.8
Mean value	23.9	Relative standard deviation	24%
Median value	25.0		

Analytical results in ascending order:

23	0.0	O	6	21.0		12	29.5	
8	5.9	O	25	24.0		13	31.2	
19	13.7		17	25.0		14	35.0	
4	17.4		7	25.0		10	47.0	O
3	18.0		1	25.3		22	94.2	O
6	19.0		15	26.7				
21	20.0		18	27.9				

O = Omitted result.

Table D.2.5. Statistics - Chloride

Sample A

Analytical method: All
Unit: mg/L

Number of participants	20	Range	0.31
Number of omitted results	2	Variance	0.00
True value	1.14	Standard deviation	0.07
Mean value	1.13	Relative standard deviation	6.0%
Median value	1.14	Relative error	-0.5%

Analytical results in ascending order:

19	0.36	O	1	1.11	23	1.15	
8	0.43	O	15	1.11	21	1.15	
3	1.02		4	1.14	2	1.16	
18	1.03		22	1.14	6	1.20	
10	1.06		7	1.14	25	1.20	
12	1.09		5	1.14	17	1.33	
13	1.10		14	1.15			

Sample B

Analytical method: All
Unit: mg/L

Number of participants	20	Range	0.21
Number of omitted results	2	Variance	0.00
True value	0.96	Standard deviation	0.06
Mean value	0.94	Relative standard deviation	6.1%
Median value	0.96	Relative error	-2.5%

Analytical results in ascending order:

19	0.16	O	15	0.92	21	0.96	
8	0.44	O	1	0.93	4	0.96	
22	0.80		7	0.95	12	0.99	
18	0.86		23	0.95	25	1.00	
3	0.87		14	0.96	6	1.00	
2	0.87		5	0.96	17	1.00	
10	0.91		13	0.96			

O = Omitted result

Table D.2.6. Statistics - Sulphate

Sample A

Analytical method: All
Unit: mg/L

Number of participants	21	Range		0.81
Number of omitted results	1	Variance		0.03
True value	1.31	Standard deviation		0.18
Mean value	1.29	Relative standard deviation		13.9%
Median value	1.31	Relative error		-1.2%

Analytical results in ascending order:

12	0.44	O	1	1.28	23	1.35
8	0.79		18	1.30	22	1.36
3	0.92		7	1.30	2	1.37
15	1.23		25	1.30	21	1.37
14	1.25		5	1.31	6	1.39
10	1.27		17	1.32	24	1.60
13	1.27		4	1.32	16	1.60

Sample B

Analytical method: All
Unit: mg/L

Number of participants	21	Range		0.65
Number of omitted results	1	Variance		0.02
True value	1.19	Standard deviation		0.15
Mean value	1.18	Relative standard deviation		12.3%
Median value	1.19	Relative error		-0.8%

Analytical results in ascending order:

12	0.40	O	14	1.16	4	1.23
8	0.80		10	1.18	22	1.24
3	0.86		1	1.18	21	1.25
15	1.12		7	1.18	6	1.25
17	1.13		25	1.20	23	1.26
2	1.13		18	1.21	24	1.40
13	1.16		5	1.22	16	1.45

O = Omitted result

Table D.2.7. Statistics - Calcium

Sample A

Analytical method: All
Unit: mg/L

Number of participants	28	Range	0.94
Number of omitted results	1	Variance	0.04
True value	1.75	Standard deviation	0.20
Mean value	1.77	Relative standard deviation	11.3%
Median value	1.75	Relative error	1.0%

Analytical results in ascending order:

3	1.16	2	1.73	7	1.90
22	1.42	23	1.73	4	1.91
1	1.52	15	1.75	10	1.94
21	1.61	14	1.75	8	1.96
19	1.61	5	1.79	12	2.05
5	1.67	16	1.79	17	2.05
23	1.68	6	1.84	13	2.10
5	1.70	25	1.86	6	2.17 O
3	1.72	20	1.87		
18	1.73	24	1.90		

Sample B

Analytical method: All
Unit: mg/L

Number of participants	28	Range	0.70
Number of omitted results	1	Variance	0.02
True value	1.63	Standard deviation	0.15
Mean value	1.62	Relative standard deviation	9.5%
Median value	1.63	Relative error	-0.8%

Analytical results in ascending order:

3	1.18	16	1.60	6	1.71
22	1.29	5	1.61	25	1.72
21	1.47	10	1.63	7	1.75
19	1.47	23	1.63	4	1.76
1	1.49	15	1.63	8	1.82
3	1.54	5	1.64	17	1.87
5	1.54	18	1.65	12	1.87
2	1.57	14	1.67	6	2.27 O
20	1.58	13	1.68		
23	1.59	24	1.70		

O = Omitted result

Table D.2.8. Statistics - Magnesium

Sample A

Analytical method: All
Unit: mg/L

Number of participants	28	Range	0.150
Number of omitted results	1	Variance	0.001
True value	0.390	Standard deviation	0.031
Mean value	0.387	Relative standard deviation	7.9%
Median value	0.390	Relative error	-0.8%

Analytical results in ascending order:

3	0.222	O	1	0.380	4	0.399
21	0.300		17	0.382	16	0.400
19	0.329		18	0.386	2	0.400
15	0.361		5	0.388	25	0.409
23	0.366		5	0.390	6	0.420
23	0.367		7	0.390	12	0.429
14	0.370		20	0.391	8	0.440
3	0.370		6	0.393	24	0.450
22	0.371		13	0.393		
5	0.377		10	0.398		

Sample B

Analytical method: All
Unit: mg/L

Number of participants	28	Range	0.101
Number of omitted results	1	Variance	0.001
True value	0.353	Standard deviation	0.024
Mean value	0.351	Relative standard deviation	6.9%
Median value	0.353	Relative error	-0.7%

Analytical results in ascending order:

3	0.198	O	20	0.344	2	0.360
19	0.299		22	0.345	18	0.361
21	0.300		24	0.350	6	0.364
3	0.310		5	0.351	4	0.364
1	0.330		17	0.353	25	0.377
15	0.334		5	0.353	6	0.390
23	0.336		13	0.356	12	0.391
14	0.340		16	0.356	8	0.400
23	0.342		5	0.357		
10	0.344		7	0.360		

O = Omitted result

Table D.2.9. Statistics - Sodium

Sample A

Analytical method: All
Unit: mg/L

Number of participants	27	Range	0.37
Number of omitted results	3	Variance	0.01
True value	1.14	Standard deviation	0.08
Mean value	1.13	Relative standard deviation	7.0%
Median value	1.14	Relative error	-0.7%

Analytical results in ascending order:

19	0.80	O	15	1.11		14	1.16	
5	0.91		1	1.12		6	1.18	
12	1.01		21	1.13		7	1.20	
10	1.05		8	1.13		4	1.21	
2	1.08		23	1.14		25	1.23	
13	1.09		16	1.14		6	1.26	
17	1.09		3	1.15		22	1.28	
18	1.10		5	1.15		24	1.70	O
20	1.10		23	1.16		3	2.70	O

Sample B

Analytical method: All
Unit: mg/L

Number of participants	27	Range	0.31
Number of omitted results	3	Variance	0.00
True value	1.00	Standard deviation	0.07
Mean value	0.99	Relative standard deviation	6.7%
Median value	1.00	Relative error	-1.3%

Analytical results in ascending order:

19	0.69	O	1	0.98		23	1.01	
5	0.78		18	0.99		13	1.01	
12	0.85		21	0.99		6	1.02	
10	0.94		3	1.00		4	1.04	
17	0.95		8	1.00		22	1.04	
2	0.96		24	1.00	O	25	1.06	
20	0.97		14	1.00		7	1.08	
16	0.97		23	1.00		6	1.09	
15	0.97		5	1.01		3	1.52	O

O = Omitted result

Table D.2.10. Statistics - Potassium

Sample A

Analytical method: All
Unit: mg/L

Number of participants	27	Range	0.080
Number of omitted results	4	Variance	0.000
True value	0.258	Standard deviation	0.019
Mean value	0.255	Relative standard deviation	7.6%
Median value	0.258	Relative error	-1.1%

Analytical results in ascending order:

3	0.168	O	16	0.255	6	0.270		
21	0.200		23	0.257	4	0.272		
13	0.222		25	0.257	5	0.272		
10	0.232		12	0.258	20	0.276		
17	0.234		2	0.260	18	0.279	O
19	0.238		7	0.260	24	0.280		
1	0.250		8	0.260	6	0.280		
14	0.250		23	0.265	3	0.290	O
5	0.254		15	0.268	22	0.525	O

Sample B

Analytical method: All
Unit: mg/L

Number of participants	27	Range	0.066
Number of omitted results	4	Variance	0.000
True value	0.220	Standard deviation	0.014
Mean value	0.218	Relative standard deviation	6.5%
Median value	0.220	Relative error	-1.1%

Analytical results in ascending order:

3	0.000	O	12	0.211	23	0.224		
3	0.152	O	10	0.211	5	0.227		
19	0.192		25	0.216	23	0.228		
21	0.200		5	0.220	6	0.230		
6	0.200		8	0.220	4	0.230		
13	0.202		7	0.220	20	0.233		
17	0.203		2	0.220	15	0.258		
24	0.210		1	0.220	18	0.291	O
14	0.210		16	0.221	22	0.302	O

O = Omitted result

Table D.2.11. Statistics - Total organic carbon

Sample A

Analytical method: All
Unit: mg/L

Number of participants	16	Range		3.56
Number of omitted results	0	Variance		0.72
True value	7.15	Standard deviation		0.85
Mean value	7.21	Relative standard deviation		11.7%
Median value	7.15	Relative error		0.8%

Analytical results in ascending order:

23	5.63	2	6.94	3	7.95
15	6.32	21	7.00	4	8.02
10	6.63	6	7.30	18	8.17
8	6.66	25	7.30	1	9.19
7	6.69	17	7.40		
16	6.70	12	7.47		

Sample B

Analytical method: All
Unit: mg/L

Number of participants	16	Range		2.44
Number of omitted results	0	Variance		0.41
True value	6.49	Standard deviation		0.64
Mean value	6.57	Relative standard deviation		9.8%
Median value	6.49	Relative error		1.2%

Analytical results in ascending order:

23	5.53	21	6.40	12	6.77
16	5.97	10	6.48	3	6.82
7	6.05	6	6.50	18	7.97
15	6.14	17	6.53	1	7.97
8	6.22	4	6.66		
2	6.36	25	6.70		

O = Omitted result

Table D.2.12. Statistics - Total phosphorous

Sample A

Analytical method: All
Unit: µg P/L

Number of participants	18	Range	9.5
Number of omitted results	8	Variance	8.1
True value	10.9	Standard deviation	2.8
Mean value	10.8	Relative standard deviation	26.2%
Median value	10.9	Relative error	-0.6%

Analytical results in ascending order:

13	0.0	O	17	10.6		23	15.5	
5	0.0	O	12	10.9		21	16.0	O
19	0.0	O	6	11.0		25	17.0	O
13	6.0		7	11.0		8	24.3	O
18	6.9		4	12.0		1	26.8	O
10	10.6		15	14.0		20	33.0	O

Sample B

Analytical method: All
Unit: µg P/L

Number of participants	18	Range	8.7
Number of omitted results	8	Variance	6.9
True value	9.1	Standard deviation	2.6
Mean value	9.1	Relative standard deviation	28.8%
Median value	9.1	Relative error	0.4%

Analytical results in ascending order:

13	0.0	O	1	8.4	O	15	12.0	
19	0.0	O	10	8.8		23	13.6	
5	3.7	O	6	9.0		25	14.0	O
18	4.9		12	9.3		21	15.0	O
13	6.0		4	9.4		20	26.0	O
17	7.4		7	11.0		8	26.5	O

O = Omitted result

Table D.2.13. Statistics - Total nitrogen

Sample A

Analytical method: All
Unit: μg N/L

Number of participants	12	Range	192
Number of omitted results	0	Variance	3221
True value	278	Standard deviation	57
Mean value	293	Relative standard deviation	19.4%
Median value	278	Relative error	5.3%

Analytical results in ascending order:

17	193	7	270	1	337
18	247	8	273	10	344
25	247	4	282	23	360
15	253	1	324	23	385

Sample B

Analytical method: All
Unit: μg N/L

Number of participants	12	Range	153
Number of omitted results	0	Variance	2289
True value	258	Standard deviation	48
Mean value	262	Relative standard deviation	18.2%
Median value	258	Relative error	1.7%

Analytical results in ascending order:

15	207	4	235	10	297
17	209	1	256	23	300
25	213	7	260	23	309
18	225	1	279	8	360

O = Omitted result

Table D.2.14. Statistics - Aluminium

Sample C

Analytical method: All

Unit: µg/L

Number of participants	17	Range	28
Number of omitted results	0	Variance	46
True value	151	Standard deviation	7
Mean value	151	Relative standard deviation	4.5%
Median value	151	Relative error	0.2%

Analytical results in ascending order:

2	140	24	150	1	154
6	143	4	150	16	154
17	143	25	151	20	155
10	147	23	153	22	162
7	148	5	153	12	168
15	148	3	153		

Sample D

Analytical method: All

Unit: µg/L

Number of participants	17	Range	31
Number of omitted results	0	Variance	48
True value	141	Standard deviation	7
Mean value	141	Relative standard deviation	4.9%
Median value	141	Relative error	0.0%

Analytical results in ascending order:

2	126	4	140	5	144
17	134	7	140	16	144
6	135	1	141	20	146
10	137	3	141	22	152
15	137	23	142	12	157
24	140	25	143		

O = Omitted result

Table D.2.15. Statistics - Iron

Sample C

Analytical method: All
Unit: µg/L

Number of participants	21	Range	21
Number of omitted results	0	Variance	34
True value	120	Standard deviation	6
Mean value	118	Relative standard deviation	5.0%
Median value	120	Relative error	-1.8%

Analytical results in ascending order:

2	105	23	117	25	121
17	106	6	117	12	122
18	111	5	120	15	122
20	113	5	120	16	122
22	114	24	120	23	124
10	115	3	120	1	126
7	116	4	121	17	126

Sample D

Analytical method: All
Unit: µg/L

Number of participants	21	Range	22
Number of omitted results	0	Variance	33
True value	110	Standard deviation	6
Mean value	107	Relative standard deviation	5.3%
Median value	110	Relative error	-2.4%

Analytical results in ascending order:

2	94	6	106	12	111
20	99	3	107	4	111
17	100	23	107	25	111
22	102	5	110	15	111
10	102	5	110	1	115
18	103	16	110	17	116
7	106	24	110	23	116

O = Omitted result

Table D.2.16. Statistics - Manganese

Sample C

Analytical method: All
Unit: µg/L

Number of participants	16	Range	0.67
Number of omitted results	1	Variance	0.03
True value	2.29	Standard deviation	0.19
Mean value	2.22	Relative standard deviation	8.4%
Median value	2.29	Relative error	-3.1%

Analytical results in ascending order:

3	1.79	20	2.27	15	2.32
2	1.80	23	2.29	4	2.33
6	2.15	24	2.30	1	2.46
17	2.19	7	2.30	22	3.20 O
5	2.23	25	2.31		
23	2.24	12	2.31		

Sample D

Analytical method: All
Unit: µg/L

Number of participants	16	Range	0.69
Number of omitted results	1	Variance	0.04
True value	2.21	Standard deviation	0.20
Mean value	2.15	Relative standard deviation	9.1%
Median value	2.21	Relative error	-2.8%

Analytical results in ascending order:

2	1.70	7	2.20	25	2.25
3	1.71	12	2.21	4	2.33
20	2.07	15	2.22	1	2.39
6	2.11	24	2.22	22	3.40 O
5	2.15	23	2.23		
17	2.18	23	2.25		

O = Omitted result

Table D.2.17. Statistics - Cadmium

Sample C

Analytical method: All
Unit: µg/L

Number of participants	16	Range		0.165
Number of omitted results	1	Variance		0.002
True value	0.650	Standard deviation		0.047
Mean value	0.640	Relative standard deviation		7.3%
Median value	0.650	Relative error		-1.6%

Analytical results in ascending order:

17	0.120	O	4	0.641	23	0.666
22	0.535		10	0.650	20	0.684
2	0.550		16	0.650	25	0.688
7	0.600		12	0.654	15	0.700
6	0.624		24	0.660		
5	0.631		3	0.660		

Sample D

Analytical method: All
Unit: µg/L

Number of participants	16	Range		0.110
Number of omitted results	1	Variance		0.001
True value	0.550	Standard deviation		0.032
Mean value	0.555	Relative standard deviation		5.7%
Median value	0.550	Relative error		0.9%

Analytical results in ascending order:

17	0.110	O	10	0.550	3	0.580
2	0.490		16	0.550	25	0.586
22	0.508		24	0.550	7	0.600
5	0.518		12	0.564	15	0.600
6	0.544		4	0.567		
20	0.549		23	0.568		

O = Omitted result

Table D.2.18. Statistics - Lead

Sample C

Analytical method: All
Unit: µg/L

Number of participants	17	Range	1.76
Number of omitted results	1	Variance	0.12
True value	2.75	Standard deviation	0.34
Mean value	2.77	Relative standard deviation	12.3%
Median value	2.75	Relative error	0.8%

Analytical results in ascending order:

7	1.90	15	2.74	24	2.90
22	2.58	5	2.74	25	2.92
2	2.60	17	2.76	20	2.96
6	2.63	23	2.78	12	3.66
3	2.72	4	2.86	1	5.01 O
16	2.73	10	2.88		

Sample D

Analytical method: All
Unit: µg/L

Number of participants	17	Range	1.36
Number of omitted results	1	Variance	0.09
True value	2.65	Standard deviation	0.30
Mean value	2.66	Relative standard deviation	11.4%
Median value	2.65	Relative error	0.5%

Analytical results in ascending order:

2	2.20	3	2.59	25	2.79
7	2.30	15	2.62	24	2.80
22	2.41	17	2.68	20	2.89
6	2.47	23	2.69	1	3.20 O
5	2.55	4	2.75	12	3.56
16	2.58	10	2.75		

O = Omitted result

Table D.2.19. Statistics - Copper

Sample C

Analytical method: All
Unit: µg/L

Number of participants	15	Range	1.52
Number of omitted results	0	Variance	0.17
True value	3.10	Standard deviation	0.41
Mean value	3.17	Relative standard deviation	13.0%
Median value	3.10	Relative error	2.3%

Analytical results in ascending order:

3	2.65	6	3.03	10	3.12
7	2.80	23	3.10	15	3.13
24	3.00	25	3.10	12	3.14
4	3.01	20	3.10	22	4.10
5	3.03	16	3.11	1	4.17

Sample D

Analytical method: All
Unit: µg/L

Number of participants	15	Range	1.13
Number of omitted results	0	Variance	0.07
True value	4.48	Standard deviation	0.26
Mean value	4.50	Relative standard deviation	5.7%
Median value	4.48	Relative error	0.5%

Analytical results in ascending order:

3	3.93	5	4.41	20	4.61
7	4.20	4	4.45	15	4.62
6	4.39	10	4.48	23	4.64
22	4.40	16	4.54	12	4.83
24	4.40	25	4.60	1	5.06

O = Omitted result

Table D.2.20. Statistics - Nickel

Sample C

Analytical method: All
Unit: µg/L

Number of participants	17		Range	1.09	
Number of omitted results	1		Variance	0.05	
True value	1.89		Standard deviation	0.23	
Mean value	1.87		Relative standard deviation	12.1%	
Median value	1.89		Relative error	-0.9%	

Analytical results in ascending order:

3	1.02	O	4	1.87	25	1.91	
2	1.20		12	1.88	15	1.97	
17	1.63		5	1.88	23	2.06	
20	1.79		7	1.90	22	2.06	
6	1.86		24	1.90	1	2.29	
10	1.87		16	1.91			

Sample D

Analytical method: All
Unit: µg/L

Number of participants	17		Range	1.10	
Number of omitted results	1		Variance	0.06	
True value	1.60		Standard deviation	0.24	
Mean value	1.63		Relative standard deviation	14.7%	
Median value	1.60		Relative error	1.6%	

Analytical results in ascending order:

3	0.72	O	10	1.57	16	1.70	
2	1.00		4	1.57	15	1.70	
17	1.41		5	1.60	23	1.86	
22	1.55		24	1.60	1	1.95	
20	1.56		25	1.64	7	2.10	
6	1.56		12	1.65			

O = Omitted result

Table D.2.21. Statistics - Zinc

Sample C

Analytical method: All
Unit: µg/L

Number of participants	19	Range	5.0
Number of omitted results	1	Variance	1.0
True value	10.6	Standard deviation	1.0
Mean value	10.5	Relative standard deviation	9.5%
Median value	10.6	Relative error	-0.5%

Analytical results in ascending order:

12	7.8	23	10.3	10	11.2
17	8.3 O	6	10.5	5	11.2
22	9.6	4	10.5	15	11.3
1	9.6	25	10.6	20	11.3
2	10.0	23	10.7	3	12.8
7	10.2	16	10.9		
5	10.2	24	11.0		

Sample D

Analytical method: All
Unit: µg/L

Number of participants	19	Range	4.7
Number of omitted results	1	Variance	1.1
True value	11.9	Standard deviation	1.1
Mean value	11.9	Relative standard deviation	8.8%
Median value	11.9	Relative error	-0.1%

Analytical results in ascending order:

17	6.9 O	6	11.7	5	12.6
12	9.2	4	11.8	16	12.7
22	10.2	1	11.9	10	12.8
2	11.1	24	12.0	15	12.8
7	11.5	23	12.1	3	13.9
23	11.5	25	12.2		
5	11.5	20	12.5		

O = Omitted result

Thematic reports from the ICP Waters programme

Since its establishment in 1985, the ICP Waters programme has prepared numerous assessments, reports and publications that address the effects of long-range transported air pollution, including thematic reports, chemical intercalibrations, biological intercalibrations, proceedings of Task Force meetings, and peer-reviewed articles. Reports and publications are available at the ICP Waters website; http://www.icp-waters.no/

Thematic reports from the ICP Waters programme from 2000 up to present are listed below.

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