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CALCULATION OF MEASUREMENT UNCERTAINTY FOR WATER MICROBIOLOGY LABORATORIES: CASE STUDY

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ABSTRACT

One of the most important tasks of water microbiology laboratories is to provide accurate and reliable analysis results when the customer undertakes. Laboratories receive accreditation certificates by applying to various national and international accreditation bodies to demonstrate that the results of the analysis are accurate and reliable on the international platform. In Turkey, this certification is carried out by the Turkish Accreditation Agency (TÜRKAK). One of the most important subjects examined under the TÜRKAK inspection in the laboratories is the measurement uncertainty of laboratories according to TS EN ISO / IEC 17025 standard. The reproducibility results are used in the calculation of measurement uncertainties in water microbiology laboratories. In *Escherichia coli* measurement uncertainty calculations, 60 cases of analysis were devised for 30 studies, supposed to be performed by two analysts (Analyst and Analyst B). As a result of our studies, RSD_R was found to be 0.075. RSD_R was considered to be combined uncertainty. For our 95% confidence interval as a result of our studies, the measurement uncertainty was calculated 15. Accurate implementation of method verifications of water microbiology laboratories will contribute to improving customer satisfaction by improving public health as it will increase quality assurance.

INTRODUCTION

One of the most important tasks of water microbiology laboratories is to provide accurate and reliable analysis results when the customer undertakes. Laboratories receive accreditation certificates by applying to various national and international accreditation bodies to demonstrate that the results of the analysis are accurate and reliable on the international platform. In Turkey, this certification is carried out by the TÜRKAK. This organization is an independent organization which is a member of the European Accreditation Association (EA), the International Accreditation Forum (IAF) and the International Laboratory Accreditation Association (ILAC). TÜRKAK conducts audits in accordance with ISO / IEC 17025 standard, which sets general conditions for testing and calibration laboratories, one of the quality management systems. One of the most important issues examined under the audit under the ISO / IEC 17025 standard is the measurement uncertainty of laboratories (1). Since standard methods are often used in water microbiology laboratories, a complete validation study is not performed. As stated in ISO / TR 13843, secondary validation, in other words, verification studies is sufficient (2). Prior to TÜRKAK's application, laboratories are responsible for proving the validity of the standard methods used by the laboratories, ie the methods for their laboratories. Method verification is the proof of the suitability of a measurement procedure for its intended use by written tests with objective testing with various performance studies The method verification also provides information on both analytical requirements (incubation temperature, food preparation and storage conditions) as well as results such as recovery (3). One of the most important criteria prior to laboratory work is the calculation of measurement uncertainty as well as starting work with competent and sufficient personnel (4). Measurement uncertainty results, which are part of the verification studies, show how accurate and reliable the analysis can be. Measurement uncertainty indicates the range of values that may be encountered with the measured value and the value supplied with the measured value. The measurement uncertainty account in microbiology laboratories is a category that prevents metrological and statistically valid computation, so it is difficult to calculate each measurement uncertainty source (5). To understand measurement uncertainty calculations in microbiology laboratories, ISO / TS 19036 (Microbiology of food and animal feeding stuffs -- Guidelines for the estimation of measurement uncertainty for quantitative determinations) is a good standard. In this standard, measurement uncertainty calculation is proposed based on experimental studies carried out in the laboratory with a black box approach that measures total variation irrespective of source (6). However, since ISO / TS 19036 is a food microbiology standard, it cannot be used in the measurement uncertainty calculations of samples in water microbiology laboratories. In water microbiology laboratories methods such as ISO / TR 13843 and ISO 17994 which provide statistical information can be used in method verification (2,7). However, laboratories may have difficulty reaching these documents because these standards have to be paid. However, the Canadian laboratory accreditation document P19 CALA provides free access (8). Another advantage of this method is that it allows the calculation of measurement uncertainty using routine samples in the laboratory. In this work, it is aimed to introduce the P19 method of the Canadian Accreditation Laboratories Association (CALA), which can be practically applied at low cost for measurement uncertainty calculations in method verification studies to accredited water microbiology laboratories according to the ISO / IEC 17025 (1).

MATERIALS AND METHODS

Data on analysis results used in measurement uncertainty calculations are randomly generated from laboratory routine studies In Escherichia coli measurement uncertainty calculations, 60 cases of analysis were devised for 30 studies, supposed to be performed by two analysts (Analyst and Analyst B). (Table1). In order for the majority of the factors that could contribute to the measurement uncertainty to be accountable, the work was carried out for a period as long as 3 months. In the reproducibility study, two analysts working on the same day were allowed to work on as many conditions as possible (different membrane filtration system, different etude, etc.) In order to reduce the measurement uncertainty, a number of experiments were performed to reduce random errors. In the calculation of the measurement uncertainty, as the first process, the reproducibility variability (S_2R) and then the reproducibility standard deviation (SD_R) were calculated. Relative standard deviation of reproducibility (RSD_R) was then calculated (Table 2). The measurement uncertainty was obtained by multiplying RSD_R by two for a 95% confidence interval.

Table 1: Measurement uncertainty study for two analysts

Date	Analyst A* - Analyst B**	Analyst A	Analyst B	Difference	Difference square
06.01.2016	Analyst A- Analyst B	88	84	4	16
06.01.2016	Analyst A- Analyst B	101	103	2	4
07.01.2016	Analyst A- Analyst B	92	87	5	25
13.01.2016	Analyst A- Analyst B	28	35	7	49
13.01.2016	Analyst A- Analyst B	58	55	3	9
14.01.2016	Analyst A- Analyst B	71	75	4	16
20.01.2016	Analyst A- Analyst B	31	28	3	9
20.01.2016	Analyst A- Analyst B	4	3	1	1
21.01.2016	Analyst A- Analyst B	16	11	5	25
27.01.2016	Analyst A- Analyst B	8	11	3	9
27.01.2016	Analyst A- Analyst B	12	18	6	36
28.01.2016	Analyst A- Analyst B	55	62	7	49
03.02.2016	Analyst A- Analyst B	82	77	5	25
03.02.2016	Analyst A- Analyst B	74	70	4	16
04.02.2016	Analyst A- Analyst B	94	90	4	16
10.02.2016	Analyst A- Analyst B	70	63	7	49
10.02.2016	Analyst A- Analyst B	16	15	1	1
11.02.2016	Analyst A- Analyst B	12	10	2	4
17.02.2016	Analyst A- Analyst B	27	24	3	9
17.02.2016	Analyst A- Analyst B	8	11	3	9
18.02.2016	Analyst A- Analyst B	18	23	5	25
24.03.2016	Analyst A- Analyst B	87	85	2	4
24.03.2016	Analyst A- Analyst B	53	52	1	1
25.02.2016	Analyst A- Analyst B	94	90	4	16
02.03.2016	Analyst A- Analyst B	58	67	9	81
02.03.2016	Analyst A- Analyst B	71	79	8	64
03.03.2016	Analyst A- Analyst B	17	24	7	49
09.03.2016	Analyst A- Analyst B	79	64	15	225
09.03.2016	Analyst A- Analyst B	35	43	8	64
10.03.2016	Analyst A- Analyst B	38	47	9	81

Analyst A*: First analyst
Analyst B**: Second analyst

RESULTS

In our measurement uncertainty study, 30 studies were constructed and 60 analyzes were conducted since both analysts worked at the same time (Table 1). As a result of our case studies, RSD_R was found to be 0.075. For the 95% confidence interval, the expanded uncertainty was calculated as 0.15, the combined uncertainty by multiplying with two. Therefore, in our study, the measurement uncertainty for *Escherichia coli* is 0.15. "U" is written as 15% when it is required to be indicated in the measurement uncertainty analysis reports.

Table 2: Measurement uncertainty calculation formulas

S^2_R=	∑(differences between pairs of analysts)^2/2N
SD_R=	$\sqrt{S^2_R}$
RSD_R=	SD_R/Mean
U	U= RSD_RX2

Table 3: Measurement uncertainty account

Total	987
Mean	50.05
S^2_R	14.51
SD_R	3.81
RSD_R	0.075
U	0.15

DISCUSSION AND CONCLUSION

In order for microbiology laboratories to become accredited, quality control studies should be carried out which include many studies that must be carried out (9). When the measurement uncertainty of the parameters in the laboratories performing water microbiology analysis is calculated, one of the most important of the quality control steps is realized. Thus, the results of the analysis are shown to be acceptable in a national and international environment. In our studies, P19 CALA, the Canadian document, was found to be a cost-effective and practically applicable method for method validation and measurement uncertainty studies in water microbiology. Realization of method verifications of water microbiology laboratories improves quality assurance. This will contribute to improving customer satisfaction as it will improve public health. For this reason, it is one of the indispensable criteria to focus training on method verifications by allocating time to personnel training, which is one of the most important points in continuous improvement studies.

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