RELACRE
ASSOCIAÇÃO DE LABORATÓRIOS ACREDITADOS DE PORTUGAL

# UNCERTAINTY FROM SAMPLING IN MICROBIOLOGICAL WATER ANALYSIS

EURACHEM WORKSHOP - Uncertainty from sampling and analysis for accredited laboratories

Berlin, 19-20 November 2019

Fátima Coimbra – RELACRE – Lisboa - Portugal

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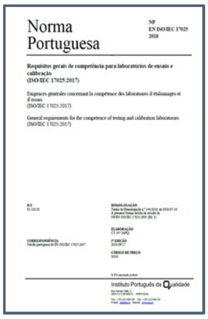
INTERNATIONAL STANDARD
ISO/IEC 17025
Third edition 2017-11

General requirements for the competence of testing and calibration laboratories

*Exigences générales concernant la compétence des laboratoires d'étalonnages et d'essais*


Norma Portuguesa
NP EN ISO/IEC 17025 2018

Requisitos gerais de competência para laboratórios de ensaio e calibração
(ISO/IEC 17025:2017)

Exigences générales concernant la compétence des laboratoires d'étalonnages et d'essais
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General requirements for the competence of testing and calibration laboratories
(ISO/IEC 17025:2017)

Instituto Português da Qualidade


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GUIA PARA A APLICAÇÃO DA NP EN ISO/IEC 17025:2018
OGC001 • 2018-12-31


INTERNATIONAL STANDARD
ISO 29201

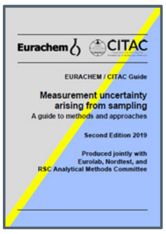
Water quality — The variability of test results and the uncertainty of measurement of microbiological enumeration methods


NT TECHNICAL REPORT

UNCERTAINTY FROM SAMPLING
– A NORDTEST HANDBOOK FOR SAMPLING PLANNERS ON SAMPLING QUALITY ASSURANCE AND UNCERTAINTY ESTIMATION
BASED UPON THE EURACHEM INTERNATIONAL GUIDE ESTIMATION OF MEASUREMENT UNCERTAINTY ARISING FROM SAMPLING

Christian Grøn, Jette Bjerre Hansen, Bertil Magnusson, Astrid Nordbotten, Mikael Krysell, Kirsten Jebjerg Andersen and Ulla Lund

NORDTEST


Eurachem CITAC

EURACHEM / CITAC Guide

Measurement uncertainty arising from sampling
A guide to methods and approaches

Second Edition 2019

Produced jointly with Eurolab, Nordtest, and RSC Analytical Methods Committee

## ISO / IEC 17025:2017

### 7.6 Evaluation of measurement uncertainty

**7.6.1** Laboratories should identify the contributions to measurement uncertainty. When evaluating measurement uncertainty, all contributions that are of significance, including those arising from sampling, shall be taken into account using appropriate methods of analysis.

**7.6.3** A laboratory performing testing shall evaluate measurement uncertainty. Where the test method precludes rigorous evaluation of measurement uncertainty, an estimation shall be made based on an understanding of the theoretical principles or practical experience of the performance of the method.

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## ISO /IEC 17025:2017

### 7.8.3 Specific requirements for test reports

**7.8.3.1** In adition to the requirements listed in 7.8.2, test reports shall, where necessary for interpretation of the tests results, include the following:

**c)** where applicable, the measurement uncertainty presented in the same units as that of the measurand or in term relative to the measurand (e.g. percent) when:

- it is relevant to the validity or application of the test results;
- a customer's instructions so requires, or
- the measurement uncertainty affects conformity to a specification limit;

## ISO /IEC 17025:2017

### 7.8.5 Reporting sampling – specific requirements

When the laboratory is responsible for the sampling activity, in addition to the requirements listed in 7.8.2, reports shall include the following, where necessary for the interpretation of the results:

f) information required to evaluate measurement uncertainty for subsequent testing or calibration.

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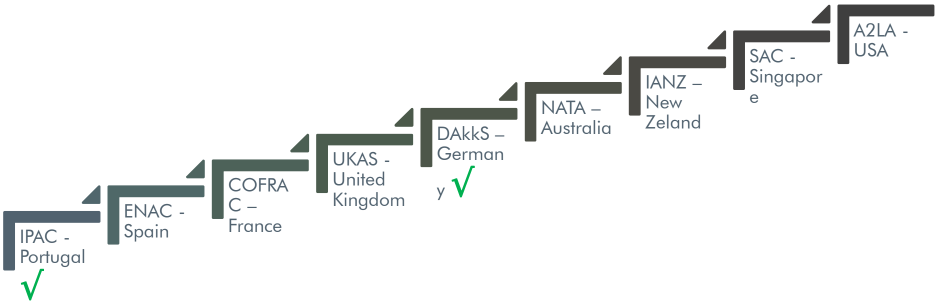
## “The state of the art in Accreditation Bodies”



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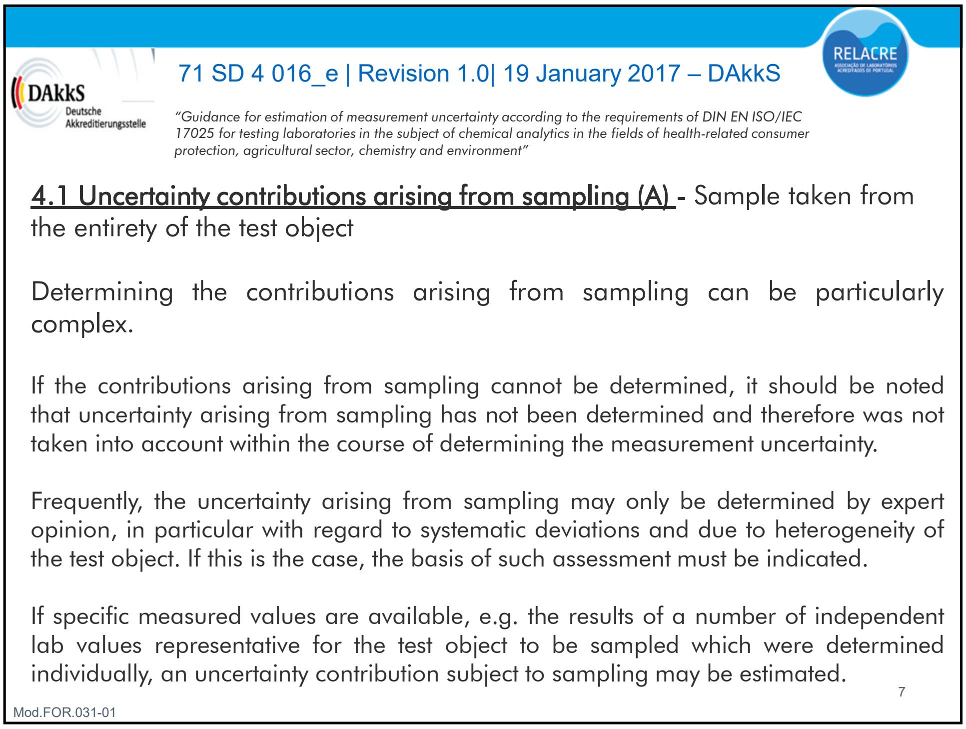
SAC - Singapore

A2LA - USA



## 71 SD 4 016_e | Revision 1.0| 19 January 2017 – DAkkS

*"Guidance for estimation of measurement uncertainty according to the requirements of DIN EN ISO/IEC 17025 for testing laboratories in the subject of chemical analytics in the fields of health-related consumer protection, agricultural sector, chemistry and environment"*

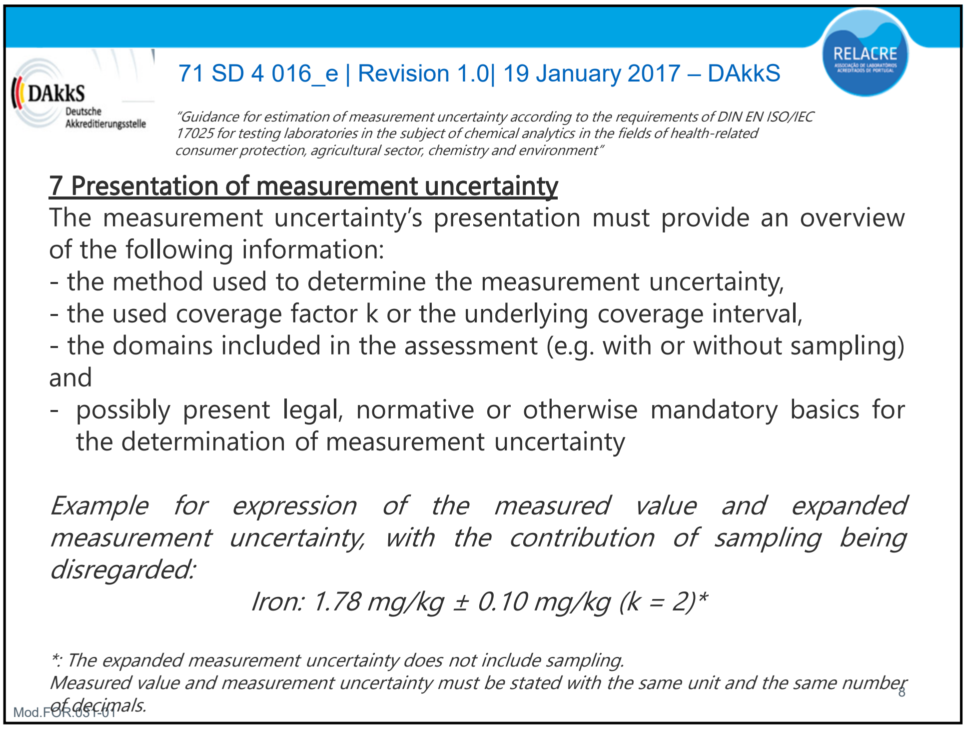
### 4.1 Uncertainty contributions arising from sampling (A) - Sample taken from the entirety of the test object

Determining the contributions arising from sampling can be particularly complex.

If the contributions arising from sampling cannot be determined, it should be noted that uncertainty arising from sampling has not been determined and therefore was not taken into account within the course of determining the measurement uncertainty.

Frequently, the uncertainty arising from sampling may only be determined by expert opinion, in particular with regard to systematic deviations and due to heterogeneity of the test object. If this is the case, the basis of such assessment must be indicated.

If specific measured values are available, e.g. the results of a number of independent lab values representative for the test object to be sampled which were determined individually, an uncertainty contribution subject to sampling may be estimated.



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### 7 Presentation of measurement uncertainty

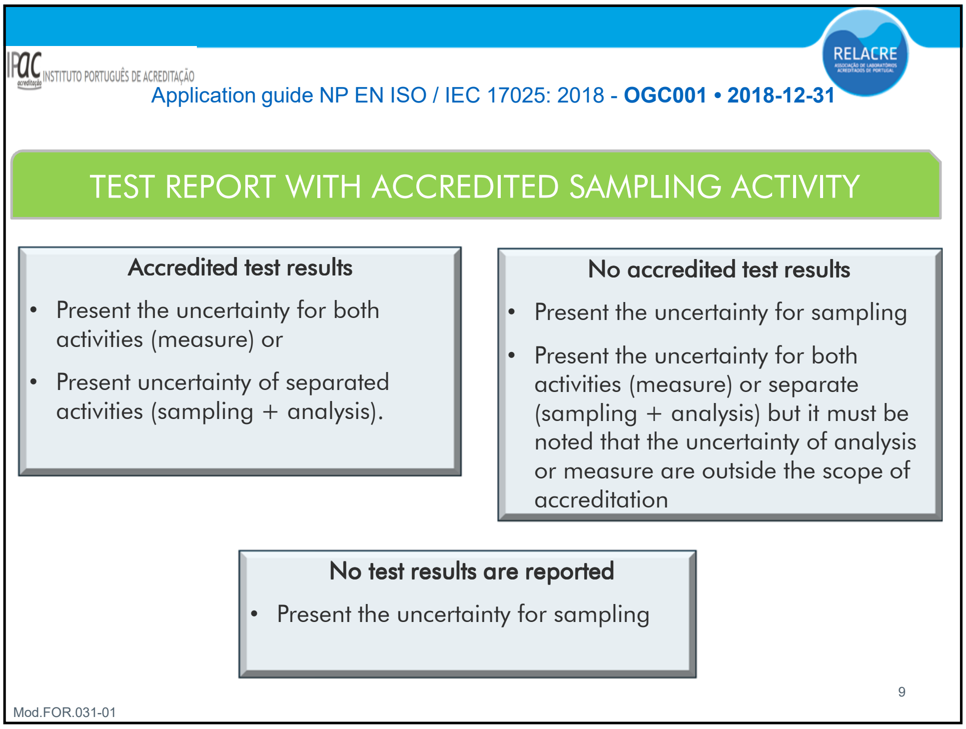
The measurement uncertainty's presentation must provide an overview of the following information:

- the method used to determine the measurement uncertainty,
- the used coverage factor k or the underlying coverage interval,
- the domains included in the assessment (e.g. with or without sampling) and
- possibly present legal, normative or otherwise mandatory basics for the determination of measurement uncertainty

*Example for expression of the measured value and expanded measurement uncertainty, with the contribution of sampling being disregarded:*

*Iron: 1.78 mg/kg ± 0.10 mg/kg (k = 2)**

**: The expanded measurement uncertainty does not include sampling.*
*Measured value and measurement uncertainty must be stated with the same unit and the same number of decimals.*



## TEST REPORT WITH ACCREDITED SAMPLING ACTIVITY

**Accredited test results**

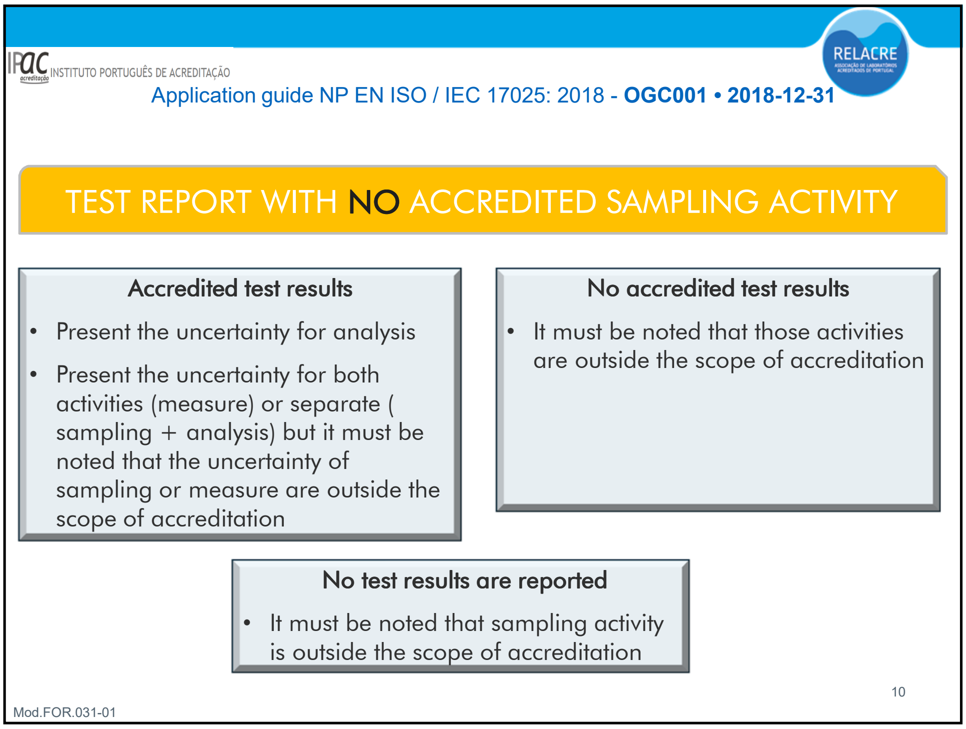
- Present the uncertainty for both activities (measure) or
- Present uncertainty of separated activities (sampling + analysis).

**No accredited test results**

- Present the uncertainty for sampling
- Present the uncertainty for both activities (measure) or separate (sampling + analysis) but it must be noted that the uncertainty of analysis or measure are outside the scope of accreditation

**No test results are reported**

- Present the uncertainty for sampling



## TEST REPORT WITH NO ACCREDITED SAMPLING ACTIVITY

**Accredited test results**

- Present the uncertainty for analysis
- Present the uncertainty for both activities (measure) or separate ( sampling + analysis) but it must be noted that the uncertainty of sampling or measure are outside the scope of accreditation

**No accredited test results**

- It must be noted that those activities are outside the scope of accreditation

**No test results are reported**

- It must be noted that sampling activity is outside the scope of accreditation

Application guide NP EN ISO / IEC 17025: 2018 - **OGC001 • 2018-12-31**

## TEST REPORT WITH NO SAMPLING ACTIVITY

**Accredited test results**

- Present the uncertainty for analysis

**No accredited test results**

- It must be noted that those results are outside the scope of accreditation

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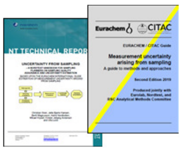
## UNCERTAINTY FROM SAMPLING IN MICROBIOLOGICAL WATER ANALISYS

INTERNATIONAL STANDARD ISO 29201

First edition 2012-01-15

__Pre-analytical sampling__ variance at the source is __outside__ the scope of this International Standard, but needs to be addressed in sampling designs and monitoring programs.



NT TECHNICAL REPORT

UNCERTAINTY FROM SAMPLING

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Measurement uncertainty arising from sampling

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Second Edition 2019

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2 Scope and field of application

2.4 Although the general principles of this Guide apply, __it does not specifically discuss microbiological sampling__.

## What is a sample?

**Sample:** A portion of material selected from a larger quantity of material.

**Representative sample:** sample resulting from a sampling plan that can be expected to reflect adequately the properties of interest in the parent population.

**Primary sample:** the collection of one or more increments or units initially taken from a population.

**Sub-sample:** Selected part of a sample.

**Laboratory sample:** sample or sub-sample sent to or received by the laboratory.

**Test sample:** sample, prepared from the laboratory sample, from which the test portions are removed for testing or for analysis.

**Test portion:** quantity of material, of proper size for measurement of the concentration or other property of interest, removed from the test sample.

## About Sampling…..

**Sampling target:** portion of material, at a particular time, that the sample is intended to represent.

1. *The sampling target should be defined prior to designing the sampling plan.*
2. *The sampling target may be defined by Regulations (e.g. lot size).*
3. *If the properties and characteristics (e.g. chemical composition) of the certain area or period are of interest and must be known then it can be considered a sampling target.*

**Sampling plan:** predetermined procedure for the selection, withdrawal, preservation, transportation and preparation of the portions to be removed from a population as a sample.

**Sampling procedure** (or protocol): operational requirements and/or instructions relating to the use of a particular sampling plan (i.e., the instructions for the implementation of the plan).

## Terms relating to sampling quality

**Replicate (duplicate) sample:** One of the two (or more*) samples or sub-samples obtained separately at the same time by the same sampling procedure or subsampling procedure.
*for replicate sample
*Note: Each duplicate sample is obtained from a separate 'sampling point' within the 'sampling location'.*

**Sampling precision:** the part of the total measurement precision attributable to the sampling.

**Sampling bias:** the part of the total measurement bias attributable to the sampling.

**Sampling uncertainty:** the part of the total measurement uncertainty attributable to the sampling

**Random sampling:** Sampling where a sample of n sampling units is taken from a population in such a way that all the possible combinations of n sampling units have a particular probability of being taken.

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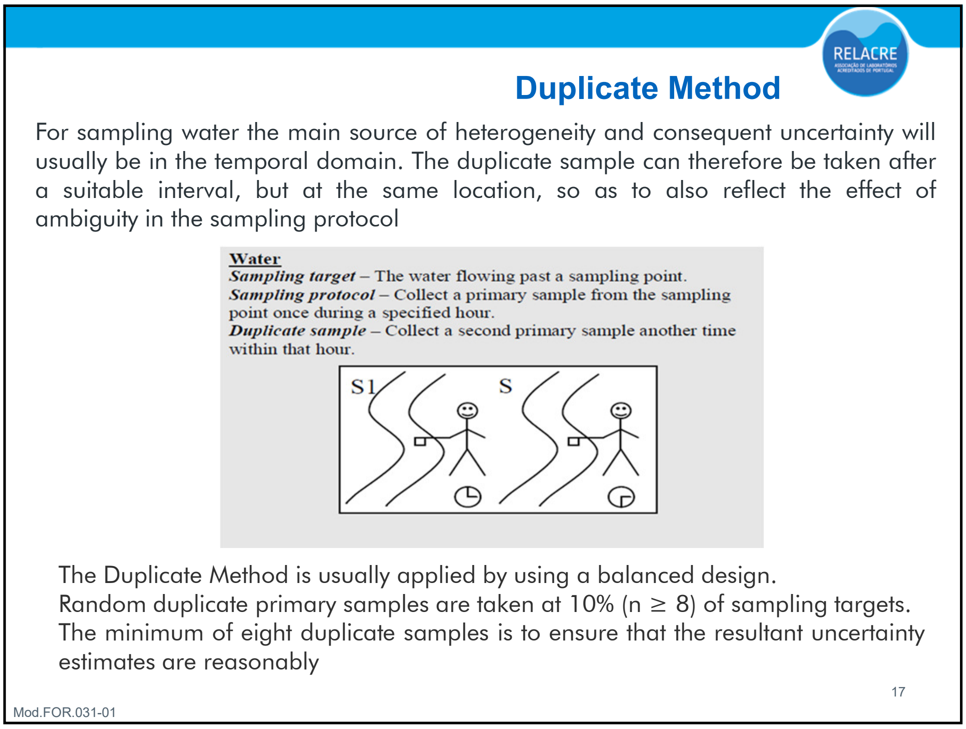
## EURACHEM / CITAC Guide 2019

1.2.3 Unlike the assumption that is often made for estimates of uncertainty for an analytical method, an estimate for one sampling protocol for one batch of material should not be assumed as automatically applicable to any subsequent batch of material.

For example, depending on the sampling target, the degree of **heterogeneity** (i.e. inhomogeneity) may have changed substantially.

There will be a need, therefore, for routine monitoring of key parameters of sampling quality to examine and update estimates of uncertainty for subsequent batches.

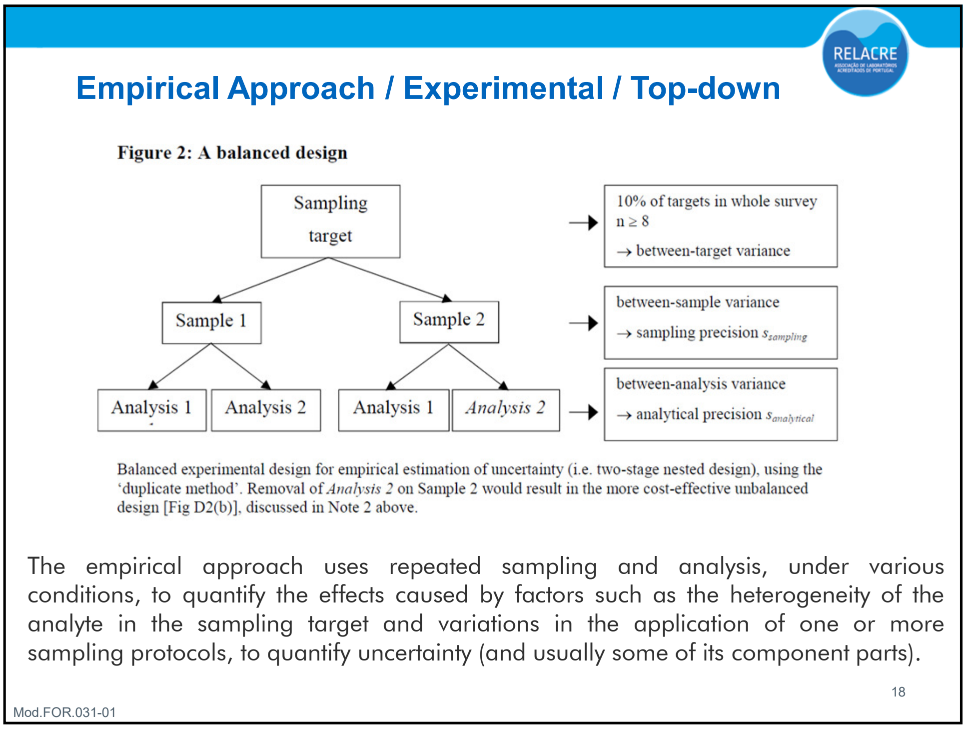


## Duplicate Method

For sampling water the main source of heterogeneity and consequent uncertainty will usually be in the temporal domain. The duplicate sample can therefore be taken after a suitable interval, but at the same location, so as to also reflect the effect of ambiguity in the sampling protocol

**Water**
***Sampling target*** – The water flowing past a sampling point.
***Sampling protocol*** – Collect a primary sample from the sampling point once during a specified hour.
***Duplicate sample*** – Collect a second primary sample another time within that hour.

The Duplicate Method is usually applied by using a balanced design.
Random duplicate primary samples are taken at 10% (n ≥ 8) of sampling targets.
The minimum of eight duplicate samples is to ensure that the resultant uncertainty estimates are reasonably



## Empirical Approach / Experimental / Top-down

**Figure 2: A balanced design**

Balanced experimental design for empirical estimation of uncertainty (i.e. two-stage nested design), using the 'duplicate method'. Removal of *Analysis 2* on Sample 2 would result in the more cost-effective unbalanced design [Fig D2(b)], discussed in Note 2 above.

The empirical approach uses repeated sampling and analysis, under various conditions, to quantify the effects caused by factors such as the heterogeneity of the analyte in the sampling target and variations in the application of one or more sampling protocols, to quantify uncertainty (and usually some of its component parts).

## Worked example

| Measurand | | | | Uncertainty estimation | | |
|---|---|---|---|---|---|---|
| Analyte/ Technique | Unit | Sector / Matrix | Sampling procedure | Purpose | Design | Statistics |
| Coliforme bacteria / Membrane Filtration - ISO 9308-1:2014 | ufc/100 mL | Microbiology / well water | Tap Water - ISO 19458:2006 - Water quality — Sampling for microbiological analysis | Uncertainty – total measurement, sampling and analytical | Empirical - duplicate method | **Robust ANOVA**<br>and<br>**New approach based on** double split design/ range statistics and **ISO 29201**<br>(both on ufc values) |

## Coliform bacteria in a well water

1) Sampling and analyses were performed by an accredited (ISO/IEC 17025) Portuguese Laboratory using accredited methods subject to the required quality assurance and analytical quality control.

2) Natural contaminated well water.

3) Ten duplicated samples C1 and C2 were taken on 10 different days.

4) Each sample C1 and C2 were test in the Laboratory in parallel ( C1.1 / C1.2 ; C2.1 / C2.2 ) different operator, the same batch of consumable, the same incubator.

5) The replicate data were treated using the range method.

6) For comparison, uncertainty estimates were calculated by analysis of variance Robust ANOVA (RANOVA) using ROBAN and a new approach based on double split design and range statistics and ISO 29201

## Coliform bacteria in a well water

| Sample n.º | Date | C1 | | C2 | |
|---|---|---|---|---|---|
| | | C1.1 | C1.2 | C2.1 | C2.2 |
| 1 | 29/05/2019 | 51 | 63 | 41 | 45 |
| 2 | 03/06/2019 | 28 | 26 | 37 | 30 |
| 3 | 04/06/2019 | 23 | 33 | 41 | 30 |
| 4 | 11/06/2019 | 5 | 18 | 13 | 18 |
| 5 | 12/06/2019 | 17 | 17 | 14 | 18 |
| 6 | 17/06/2019 | 39 | 68 | 39 | 67 |
| 7 | 18/06/2019 | 53 | 62 | 42 | 46 |
| 8 | 19/06/2019 | 29 | 27 | 17 | 19 |
| 9 | 24/06/2019 | 24 | 36 | 41 | 32 |
| 10 | 25/06/2019 | 54 | 61 | 44 | 48 |

- Measurements of the number of colonies (ufc/100mL) of Coliform bacteria in ten duplicated samples.
- The duplicate samples are labelled C1 and C2. Likewise, duplicate analyses are labelled C1.1/ C1.2 and C2.1 and C2.2

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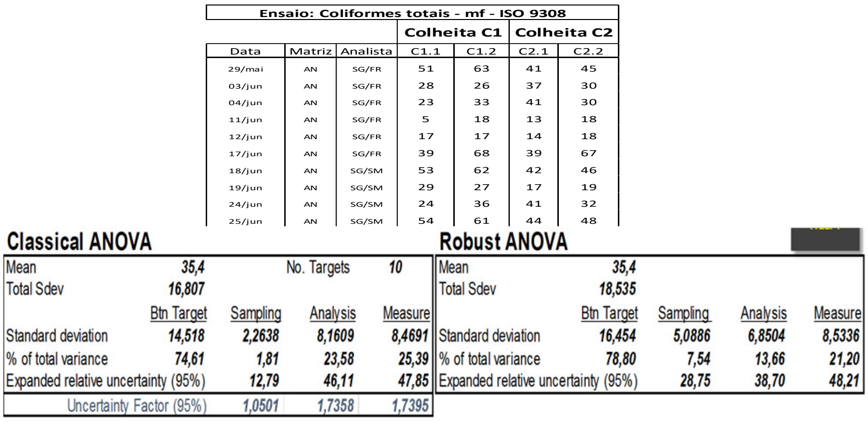
## Output of Classical and Robust ANOVA



| Ensaio: Coliformes totais - mf - ISO 9308 | | | | | | |
|---|---|---|---|---|---|---|
| | | | Colheita C1 | | Colheita C2 | |
| Data | Matriz | Analista | C1.1 | C1.2 | C2.1 | C2.2 |
| 29/mai | AN | SG/FR | 51 | 63 | 41 | 45 |
| 03/jun | AN | SG/FR | 28 | 26 | 37 | 30 |
| 04/jun | AN | SG/FR | 23 | 33 | 41 | 30 |
| 11/jun | AN | SG/FR | 5 | 18 | 13 | 18 |
| 12/jun | AN | SG/FR | 17 | 17 | 14 | 18 |
| 17/jun | AN | SG/FR | 39 | 68 | 39 | 67 |
| 18/jun | AN | SG/SM | 53 | 62 | 42 | 46 |
| 19/jun | AN | SG/SM | 29 | 27 | 17 | 19 |
| 24/jun | AN | SG/SM | 24 | 36 | 41 | 32 |
| 25/jun | AN | SG/SM | 54 | 61 | 44 | 48 |

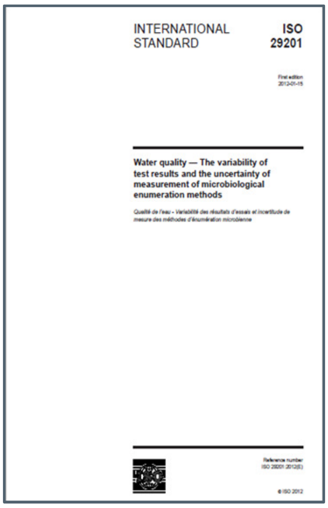
**Classical ANOVA**

| | | | | |
|---|---|---|---|---|
| Mean | 35,4 | | No. Targets | 10 |
| Total Sdev | 16,807 | | | |
| | Btn Target | Sampling | Analysis | Measure |
| Standard deviation | 14,518 | 2,2638 | 8,1609 | 8,4691 |
| % of total variance | 74,61 | 1,81 | 23,58 | 25,39 |
| Expanded relative uncertainty (95%) | | 12,79 | 46,11 | 47,85 |
| Uncertainty Factor (95%) | | 1,0501 | 1,7358 | 1,7395 |

**Robust ANOVA**

| | | | | |
|---|---|---|---|---|
| Mean | 35,4 | | | |
| Total Sdev | 18,535 | | | |
| | Btn Target | Sampling | Analysis | Measure |
| Standard deviation | 16,454 | 5,0886 | 6,8504 | 8,5336 |
| % of total variance | 78,80 | 7,54 | 13,66 | 21,20 |
| Expanded relative uncertainty (95%) | | 28,75 | 38,70 | 48,21 |

## ISO 29201:2012 Water Quality – The variability of test results and uncertainty of measurent of microbiologycal enumeration methods



INTERNATIONAL STANDARD

ISO 29201

First edition
2012-01-15

**Water quality — The variability of test results and the uncertainty of measurement of microbiological enumeration methods**

*Qualité de l'eau - Variabilité des résultats d'essais et incertitude de mesure des méthodes d'énumération microbienne*

Reference number
ISO 29201:2012(E)

© ISO 2012

IMPORTANT NOTES

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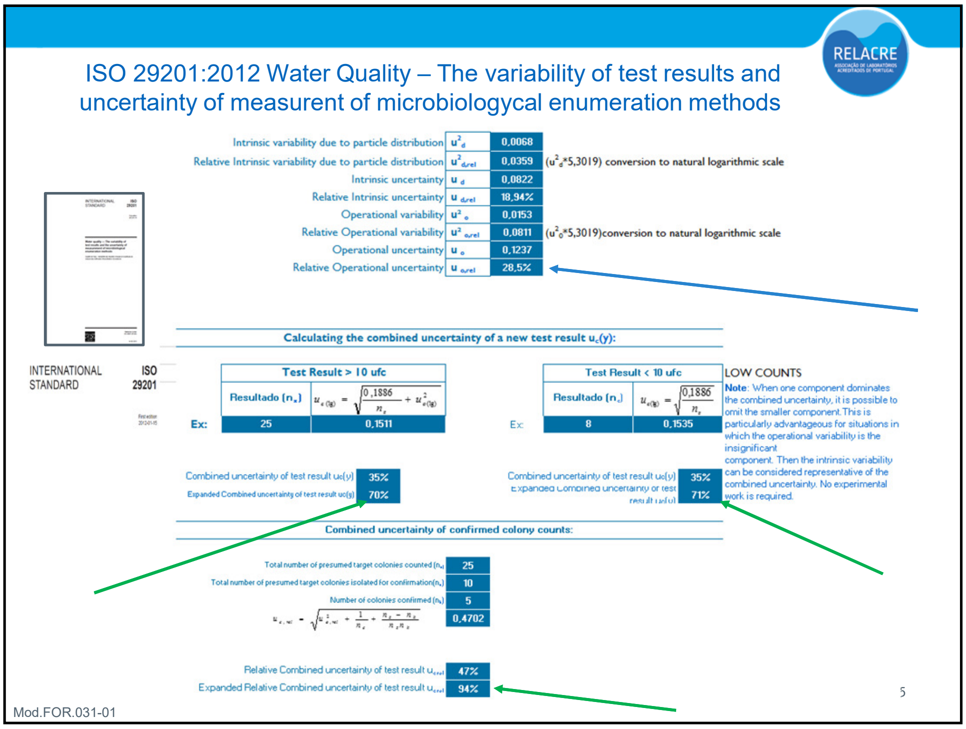
## ISO 29201:2012 Water Quality – The variability of test results and uncertainty of measurent of microbiologycal enumeration methods

### Expression and use of measurement uncertainty

When working according to this International Standard, the laboratory should be able to obtain an estimate of the operational uncertainty**,** $u_o$, for **every relevant method and sample type combination** under intralaboratory reproducibility (intermediate precision) conditions.

### Combined uncertainty of measurement of a test result

When requested by customers or accreditors, an estimate of the **combined uncertainty of measurement** of a **test result** is constructed from the **test result**, $n_z$, and the **relative operational uncertainty,** $u_o$ **rel.**



## ISO 29201:2012 Water Quality – The variability of test results and uncertainty of measurent of microbiologycal enumeration methods

INTERNATIONAL STANDARD ISO 29201

First edition 2012-01-15

| | | | |
|---|---|---|---|
| Intrinsic variability due to particle distribution | $u^2_d$ | 0,0068 | |
| Relative Intrinsic variability due to particle distribution | $u^2_{d,rel}$ | 0,0359 | ($u^2_d$*5,3019) conversion to natural logarithmic scale |
| Intrinsic uncertainty | $u_d$ | 0,0822 | |
| Relative Intrinsic uncertainty | $u_{d,rel}$ | 18,94% | |
| Operational variability | $u^2_o$ | 0,0153 | |
| Relative Operational variability | $u^2_{o,rel}$ | 0,0811 | ($u^2_o$*5,3019)conversion to natural logarithmic scale |
| Operational uncertainty | $u_o$ | 0,1237 | |
| Relative Operational uncertainty | $u_{o,rel}$ | 28,5% | |

**Calculating the combined uncertainty of a new test result $u_c(y)$:**

| | Test Result > 10 ufc | |
|---|---|---|
| | Resultado ($n_x$) | $u_{c(lg)} = \sqrt{\frac{0,1886}{n_z} + u^2_{o(lg)}}$ |
| Ex: | 25 | 0,1511 |

| | | |
|---|---|---|
| Combined uncertainty of test result uc(y) | 35% |
| Expanded Combined uncertainty of test result uc(y) | 70% |

| | Test Result < 10 ufc | |
|---|---|---|
| | Resultado ($n_x$) | $u_{c(lg)} = \sqrt{\frac{0,1886}{n_z}}$ |
| Ex: | 8 | 0,1535 |

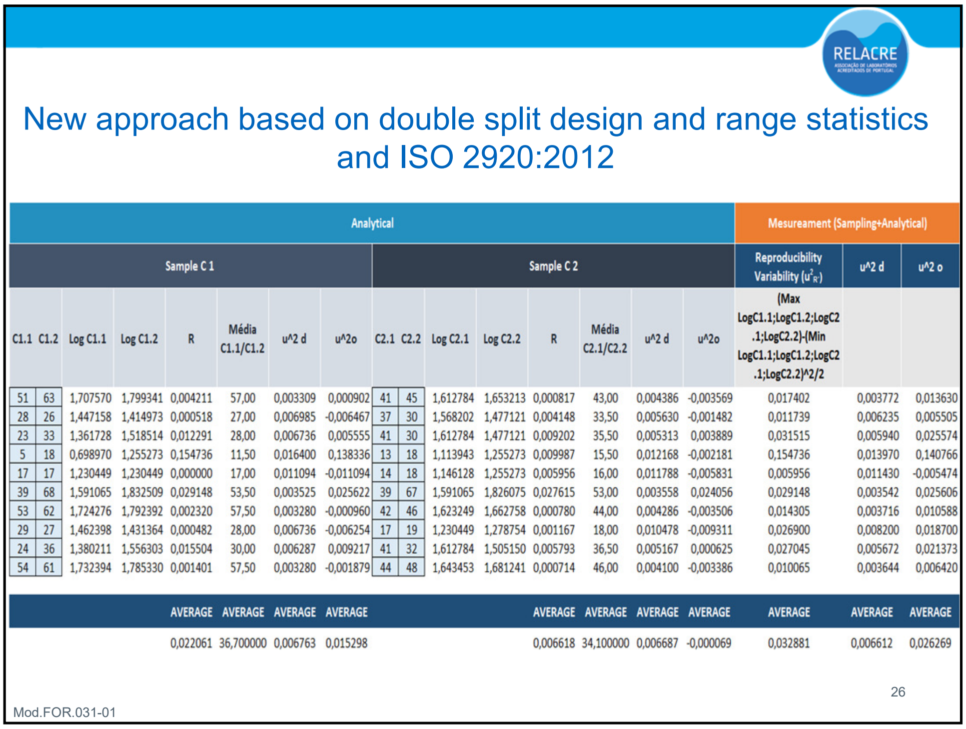
| | |
|---|---|
| Combined uncertainty of test result uc(y) | 35% |
| Expanded Combined uncertainty of test result uc(y) | 71% |

LOW COUNTS

**Note**: When one component dominates the combined uncertainty, it is possible to omit the smaller component. This is particularly advantageous for situations in which the operational variability is the insignificant component. Then the intrinsic variability can be considered representative of the combined uncertainty. No experimental work is required.

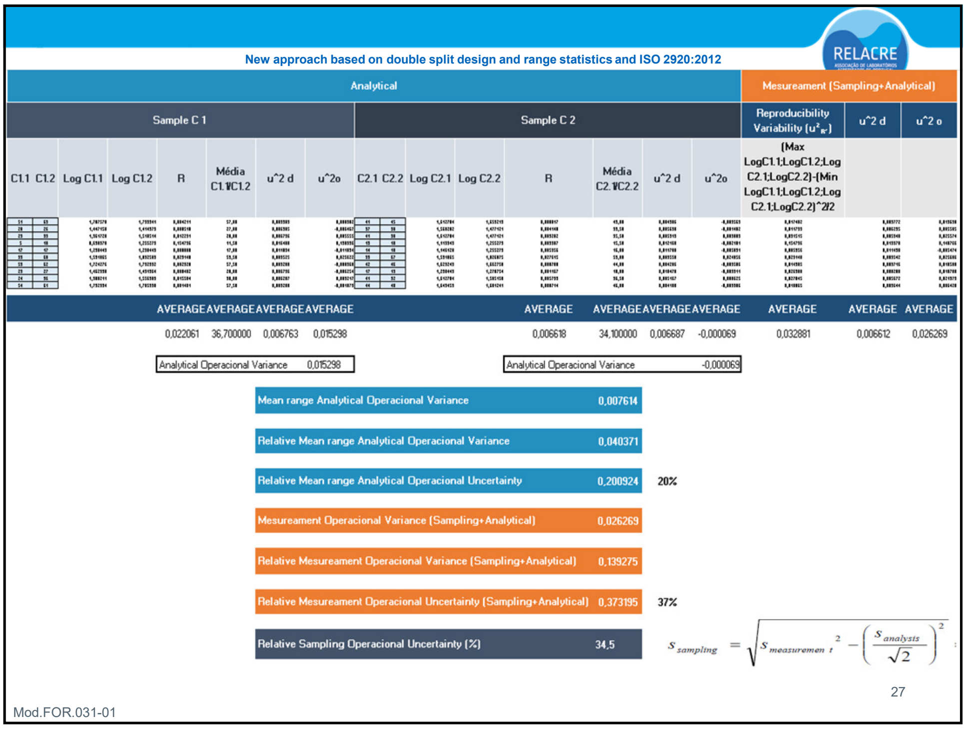
**Combined uncertainty of confirmed colony counts:**

| | |
|---|---|
| Total number of presumed target colonies counted ($n_z$) | 25 |
| Total number of presumed target colonies isolated for confirmation($n_t$) | 10 |
| Number of colonies confirmed ($n_k$) | 5 |
| $u_{c,rel} = \sqrt{u^2_{c,rel} + \frac{1}{n_c} + \frac{n_t - n_k}{n_t n_k}}$ | 0,4702 |
| Relative Combined uncertainty of test result $u_{c,rel}$ | 47% |
| Expanded Relative Combined uncertainty of test result $u_{c,rel}$ | 94% |



## New approach based on double split design and range statistics and ISO 2920:2012

| Analytical | | | | | | | | | | | | | | | | Mesureament (Sampling+Analytical) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample C 1 | | | | | | | | Sample C 2 | | | | | | | | Reproducibility Variability ($u^2_{R'}$) | u^2 d | u^2 o |
| C1.1 | C1.2 | Log C1.1 | Log C1.2 | R | Média C1.1/C1.2 | u^2 d | u^2o | C2.1 | C2.2 | Log C2.1 | Log C2.2 | R | Média C2.1/C2.2 | u^2 d | u^2o | (Max LogC1.1;LogC1.2;LogC2.1;LogC2.2)-(Min LogC1.1;LogC1.2;LogC2.1;LogC2.2)^2/2 | | |
| 51 | 63 | 1,707570 | 1,799341 | 0,004211 | 57,00 | 0,003309 | 0,000902 | 41 | 45 | 1,612784 | 1,653213 | 0,000817 | 43,00 | 0,004386 | -0,003569 | 0,017402 | 0,003772 | 0,013630 |
| 28 | 26 | 1,447158 | 1,414973 | 0,000518 | 27,00 | 0,006985 | -0,006467 | 37 | 30 | 1,568202 | 1,477121 | 0,004148 | 33,50 | 0,005630 | -0,001482 | 0,011739 | 0,006235 | 0,005505 |
| 23 | 33 | 1,361728 | 1,518514 | 0,012291 | 28,00 | 0,006736 | 0,005555 | 41 | 30 | 1,612784 | 1,477121 | 0,009202 | 35,50 | 0,005313 | 0,003889 | 0,031515 | 0,005940 | 0,025574 |
| 5 | 18 | 0,698970 | 1,255273 | 0,154736 | 11,50 | 0,016400 | 0,138336 | 13 | 18 | 1,113943 | 1,255273 | 0,009987 | 15,50 | 0,012168 | -0,002181 | 0,154736 | 0,013970 | 0,140766 |
| 17 | 17 | 1,230449 | 1,230449 | 0,000000 | 17,00 | 0,011094 | -0,011094 | 14 | 18 | 1,146128 | 1,255273 | 0,005956 | 16,00 | 0,011788 | -0,005831 | 0,005956 | 0,011430 | -0,005474 |
| 39 | 68 | 1,591065 | 1,832509 | 0,029148 | 53,50 | 0,003525 | 0,025622 | 39 | 67 | 1,591065 | 1,826075 | 0,027615 | 53,00 | 0,003558 | 0,024056 | 0,029148 | 0,003542 | 0,025606 |
| 53 | 62 | 1,724276 | 1,792392 | 0,002320 | 57,50 | 0,003280 | -0,000960 | 42 | 46 | 1,623249 | 1,662758 | 0,000780 | 44,00 | 0,004286 | -0,003506 | 0,014305 | 0,003716 | 0,010588 |
| 29 | 27 | 1,462398 | 1,431364 | 0,000482 | 28,00 | 0,006736 | -0,006254 | 17 | 19 | 1,230449 | 1,278754 | 0,001167 | 18,00 | 0,010478 | -0,009311 | 0,026900 | 0,008200 | 0,018700 |
| 24 | 36 | 1,380211 | 1,556303 | 0,015504 | 30,00 | 0,006287 | 0,009217 | 41 | 32 | 1,612784 | 1,505150 | 0,005793 | 36,50 | 0,005167 | 0,000625 | 0,027045 | 0,005672 | 0,021373 |
| 54 | 61 | 1,732394 | 1,785330 | 0,001401 | 57,50 | 0,003280 | -0,001879 | 44 | 48 | 1,643453 | 1,681241 | 0,000714 | 46,00 | 0,004100 | -0,003386 | 0,010065 | 0,003644 | 0,006420 |
| | | | | AVERAGE | AVERAGE | AVERAGE | AVERAGE | | | | | AVERAGE | AVERAGE | AVERAGE | AVERAGE | AVERAGE | AVERAGE | AVERAGE |
| | | | | 0,022061 | 36,700000 | 0,006763 | 0,015298 | | | | | 0,006618 | 34,100000 | 0,006687 | -0,000069 | 0,032881 | 0,006612 | 0,026269 |



New approach based on double split design and range statistics and ISO 2920:2012

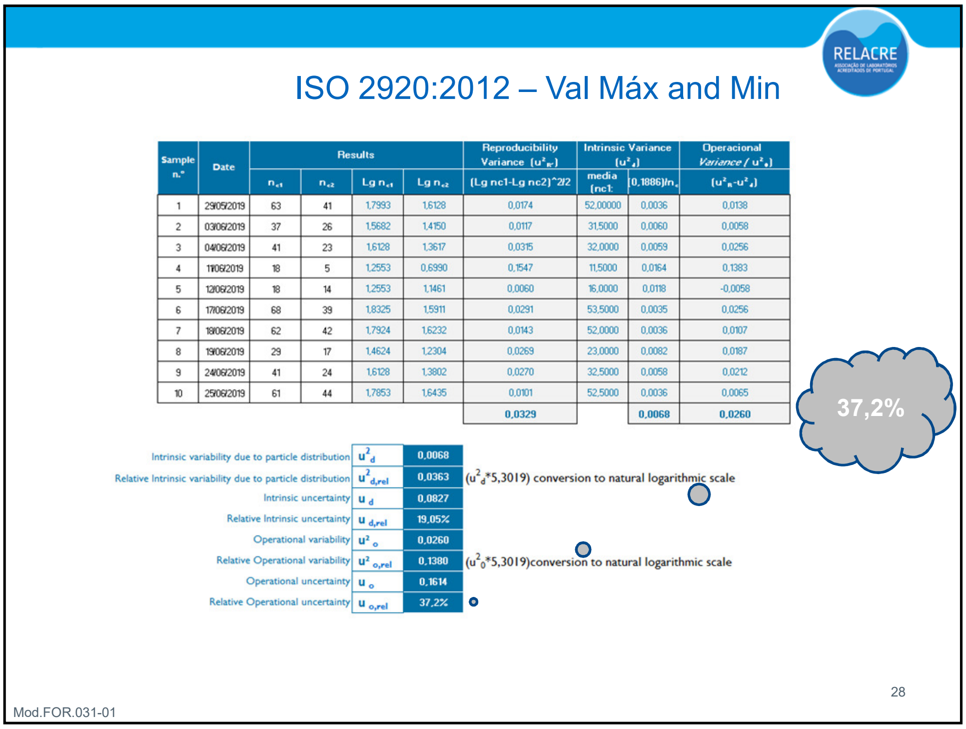
| Analytical | | | | | | | | | | | | | | | | Mesureament (Sampling+Analytical) | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample C 1 | | | | | | | | Sample C 2 | | | | | | | | Reproducibility Variability ($u^2_{R'}$) | u^2 d | u^2 o |
| C1.1 | C1.2 | Log C1.1 | Log C1.2 | R | Média C1.1/C1.2 | u^2 d | u^2o | C2.1 | C2.2 | Log C2.1 | Log C2.2 | R | Média C2.1/C2.2 | u^2 d | u^2o | (Max LogC1.1;LogC1.2;Log C2.1;LogC2.2)-(Min LogC1.1;LogC1.2;Log C2.1;LogC2.2)^2/2 | | |
| | | | | AVERAGE | AVERAGE | AVERAGE | AVERAGE | | | | | AVERAGE | AVERAGE | AVERAGE | AVERAGE | AVERAGE | AVERAGE | AVERAGE |
| | | | | 0,022061 | 36,700000 | 0,006763 | 0,015298 | | | | | 0,006618 | 34,100000 | 0,006687 | -0,000069 | 0,032881 | 0,006612 | 0,026269 |

| | |
|---|---|
| Analytical Operacional Variance | 0,015298 |

| | |
|---|---|
| Analytical Operacional Variance | -0,000069 |

| | | |
|---|---|---|
| Mean range Analytical Operacional Variance | 0,007614 | |
| Relative Mean range Analytical Operacional Variance | 0,040371 | |
| Relative Mean range Analytical Operacional Uncertainty | 0,200924 | 20% |
| Mesureament Operacional Variance (Sampling+Analytical) | 0,026269 | |
| Relative Mesureament Operacional Variance (Sampling+Analytical) | 0,139275 | |
| Relative Mesureament Operacional Uncertainty (Sampling+Analytical) | 0,373195 | 37% |
| Relative Sampling Operacional Uncertainty (%) | 34,5 | |

$$s_{sampling} = \sqrt{s_{measurement}^{2} - \left(\frac{s_{analysis}}{\sqrt{2}}\right)^{2}}$$



# ISO 2920:2012 – Val Máx and Min

| Sample n.º | Date | Results | | | | Reproducibility Variance ($u^2_{R'}$) | Intrinsic Variance ($u^2_d$) | | Operacional *Variance / $u^2_o$* |
|---|---|---|---|---|---|---|---|---|---|
| | | $n_{c1}$ | $n_{c2}$ | Lg $n_{c1}$ | Lg $n_{c2}$ | (Lg nc1-Lg nc2)^2/2 | media (nc1: | (0,1886)/$n_c$ | ($u^2_R$-$u^2_d$) |
| 1 | 29/05/2019 | 63 | 41 | 1,7993 | 1,6128 | 0,0174 | 52,00000 | 0,0036 | 0,0138 |
| 2 | 03/06/2019 | 37 | 26 | 1,5682 | 1,4150 | 0,0117 | 31,5000 | 0,0060 | 0,0058 |
| 3 | 04/06/2019 | 41 | 23 | 1,6128 | 1,3617 | 0,0315 | 32,0000 | 0,0059 | 0,0256 |
| 4 | 11/06/2019 | 18 | 5 | 1,2553 | 0,6990 | 0,1547 | 11,5000 | 0,0164 | 0,1383 |
| 5 | 12/06/2019 | 18 | 14 | 1,2553 | 1,1461 | 0,0060 | 16,0000 | 0,0118 | -0,0058 |
| 6 | 17/06/2019 | 68 | 39 | 1,8325 | 1,5911 | 0,0291 | 53,5000 | 0,0035 | 0,0256 |
| 7 | 18/06/2019 | 62 | 42 | 1,7924 | 1,6232 | 0,0143 | 52,0000 | 0,0036 | 0,0107 |
| 8 | 19/06/2019 | 29 | 17 | 1,4624 | 1,2304 | 0,0269 | 23,0000 | 0,0082 | 0,0187 |
| 9 | 24/06/2019 | 41 | 24 | 1,6128 | 1,3802 | 0,0270 | 32,5000 | 0,0058 | 0,0212 |
| 10 | 25/06/2019 | 61 | 44 | 1,7853 | 1,6435 | 0,0101 | 52,5000 | 0,0036 | 0,0065 |
| | | | | | | **0,0329** | | **0,0068** | **0,0260** |

37,2%

| | | | |
|---|---|---|---|
| Intrinsic variability due to particle distribution | $u^2_d$ | 0,0068 | |
| Relative Intrinsic variability due to particle distribution | $u^2_{d,rel}$ | 0,0363 | ($u^2_d$*5,3019) conversion to natural logarithmic scale |
| Intrinsic uncertainty | $u_d$ | 0,0827 | |
| Relative Intrinsic uncertainty | $u_{d,rel}$ | 19,05% | |
| Operational variability | $u^2_o$ | 0,0260 | |
| Relative Operational variability | $u^2_{o,rel}$ | 0,1380 | ($u^2_0$*5,3019)conversion to natural logarithmic scale |
| Operational uncertainty | $u_o$ | 0,1614 | |
| Relative Operational uncertainty | $u_{o,rel}$ | 37,2% | |

## And Now?!

**Sampling Uncertainty Working Group (Eurachem)** - prepare guidance for the evaluation of uncertainties arinsing from the process of sampling applicable to all microbiological mesurements ( water and food) that require the taking of a sample.

**Accreditation Bodies** - assessing peer policies together while the scientific community lacks a validated model for estimating uncertainty from sampling in microbiological measurements (water and food).

## References

ISO / IEC 17025: 2017 – General requirements for the competence of testing and calibration laboratories

ISO 29201:2012 –Water Quality – The variability of test results and uncertainty of measurent of microbiological enumeration methods

ISO 19458:2006 - Water quality — Sampling for microbiological analysis

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https://www.nordtest.info/images/documents/nt-technical-TechnicalReport.pdf

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Thank you for your attention

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