PROFICIENCY TEST
QUALINOVA

QUALINOVA XXXXXXX

CIRCUIT XXXXX

ROUND: XXXXX

Date of issue: XX-XX-XXXX

Review 0

QUALINOVA
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SIGN	SIGN
NAME	NAME
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Date of issue: XXXXXXX

The Proficiency Tests marketed under the brand name QUALINOVA are the responsibility of Labnova Distribuciones Agroalimentarias S.L.

All information reported by participants and included in this report is used confidentially by the proficiency testing programme provider.

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In this report we present you the results of the PT round XXXXX from the program Qualinova XXXX in which one you had participated.

The analysis of the results and the statistical treatment had been carried out on the data provided by the participants in computer support, without any other operation of transcription or manipulation thereof.

All the information reported by the participants and included in this report, is used in a confidential way by the provider of the proficiency tests programs.

1. GENERAL ASPECTS

The Proficiency Tests marketed under the QUALINOVA brand are the responsibility of Labnova Distribuciones Agroalimentarias S.L.

The organization and development of the Qualinova Proficiency Tests is based on the following documents:

- UNE-EN ISO/IEC 17043 "*Evaluación de la conformidad. Requisitos generales para la competencia de los proveedores de ensayos de aptitud*".
- ISO 13528 "*Statistical methods for use in proficiency testing by interlaboratory comparison".*
- UNE-EN ISO/IEC 17025 "*Requisitos generales para la competencia de los laboratorios de ensayo y calibración".*
- G-ENAC-14 "*Directrices sobre la participación en programas de ensayos de aptitud*".
- Eurachem guide of *Selection, use and interpretation of PT schemes*.
- IUPAC 2006 *International harmonized protocol for the proficiency testing.*
- Thomson, Michael: *Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing.* The Analyst communication. Enero 2000.
- Huber, P. J. (1981). *Robust Statistics*, Wiley, N. Y.
- ISO standards that may be used for specific matrixes or parameters.

The management of the Proficiency Test Circuit rounds organized by Qualinova is independent, and it is not necessary to register in full or even a minimum number. The general structure of each of the rounds that make up the Qualinova Proficiency Tests Programs is as follows:

- The Qualinova organization is responsible for preparing and distributing the prepared samples according to internal procedures. Each sample is identified with a label that includes the program, circuit, round code and contents of the container.

- The organization provides via email the instructions to prepare the samples and carry out the corresponding tests. It is necessary to preserve and treat the sample according to the instructions received.

- In each round the participant can perform all the proposed analysis or only those of his interest.

- Participating laboratories must communicate the results obtained through the website www.labnovasl.com/qualinova, accessing the customer portal with the username and password provided by the organization.

- Entered values must be indicated with a point as thousands separators (.), and decimals must be separated by a comma (,).

- Microbiology results must be expressed in the requested units, without exponential expressions such as 7E03 or powers of 10. The figures must be integers: 7,000, 1,000,000, etc.

- Each participant must indicate the used method for each analysis in the corresponding box of the results registration form.

- After the reception of all the results, the Qualinova organization carries out the statistical treatment of the results and drafts the final report of the round, along with the the personalized reports for each participant.

- The participating laboratories will receive the report where their performance is evaluated. Each laboratory is identified with a private code, so the confidentiality of the results is ensured. The code is indicated in the personalized report that accompanies the general report.

- In case that a participant wants to file a complaint or appeal regarding the evaluation of their performance in a round, they must contact the email address qualinova@labnovasl.com, where their request will be studied and assessed.

1.1. Definitions.

Proficiency tests provider: Organization which takes responsibility for all tasks in the development and operation of a *proficiency testing* scheme.

Interlaboratory comparison: Organization, performance and evaluation of measurements or test on the same or similar items by wo or more laboratories in accordance with predetermined conditions.

Proficiency testing (PT): Evaluation of participant performance against pre-established criteria by means of *interlaboratory comparisons*.

Proficiency test item: Sample, product, artefact, reference material, piece of equipment, measurement standard, data set or other information used to assess *participant* performance in *proficiency testing.*

Assigned value (X_t): Value attributed to a particular property of a proficiency test item.

Consensus value: Value derived from a collection of results in an *interlaboratory comparison*.

Outlier: Member of a set of values which is inconsistent with other members of that set.

Robust mean ($\bar{x}^*$): Average of all values, obtained through the application of robust techniques that minimize the effect of extreme values. In general, this value is obtained by applying winsorization using the Huber H15 method.

Robust standard deviation (S*): Value of the dispersion of the results of the exercise calculated using robust techniques, to minimize the effect of extreme values. It is also obtained through the Huber H15 system.

Standard deviation for proficiency assessment (σ_{pt}): Measure of dispersion used in evaluating the results of a proficiency test, based on available information. Also known as "target" or "objective" deviation.

Uncertainty of assigned value (μ_x): Uncertainty associated with the method of calculating the assigned value.

Z-score: Standardized measure of performance, calculated using the participant's result, assigned value, and standard deviation for aptitude evaluation.

1.2. Standard deviation for proficiency assessment.

According to the ISO 13528 standard, these are the possible approximations to estimate (σ_{pt}):

A. Perception by experts. The maximum permissible error of (σ_{pt}) can be established as the value that corresponds to the level of performance that a regulatory authority, accreditation body or technical experts of the Proficiency Testing provider consider to be reasonable for the participants.

B. Due to the experience acquired in previous rounds of an aptitude test.

C. Use of a general model, usually used in the chemical field, is the one described by Horwitz and modified by Thompson. This approach provides a general model for the reproducibility of

analytical methods that can be used to produce mathematical expressions that allow calculating the standard deviation of reproducibility.

D. Use of the standard deviation of repeatability and reproducibility obtained in previous collaborative studies of the precision of a measurement method.

E. Use of the data obtained in the Proficiency Test round itself. It should be kept in mind that the use of participants' own results may result in criteria for performance evaluation that may not be appropriate. The Proficiency Test provider must always ensure that the (σ_{pt}) used is suitable for the intended purpose.

In the case of microbiological parameters, a combination of approaches A and B is followed, based on the experience of the Expert of the Proficiency Testing provider, in rounds of previous years and in point 8.3.6.2 of the UNE-EN ISO standard. 22117: "*The z-score calculation uses an ideal standard deviation value (σpt). This ideal standard deviation defines the acceptable range of variation between laboratories for a particular assay, with σpt values of 0,35 or 0,25 commonly used in microbiological AE schemes*."

In the case of physical-chemical parameters, whenever there is regulation or legislation with data on tolerances, uncertainties or precision, these data will be taken to set the σ_{pt} values. If there is no legislation, the Expert of the Proficiency Testing provider will be consulted and historical data will be reviewed. As the next option, the general Horwitz model modified by Thompson will be used.

1.3. Homogeneity and stability.

A homogeneity and stability study of the prepared samples is carried out, in accordance with the procedures established by ISO 17043 and ISO 13528, maintaining the samples in the same conditions in which they will be received in the destination laboratories. The parameters analysis are developed by a laboratory accredited under the ISO 17025 Standard.

To carry out the homogeneity study, 10 samples are taken at random from all the prepared vials and two portions of each are analysed under repeatability conditions. It is checked whether the homogeneity criterion established in the ISO 13528 Standard is met, by which the standard deviation between samples (S_s) must be less than or equal to 0,3 times the "target" deviation (σ_{pt}) established for each of the parameters.

If this criterion is not met, the criterion indicated in section B.2.3 of the ISO 13528 Standard is verified – *It may be useful to expand the criterion to allow for the actual sampling error and repeatability in the homogeneity check.* The sample is accepted as homogeneous as long as: $s_s < \sqrt{c}$, where $c = F_1\sigma_{allow}^2 + F_2 s_w^2$ where the factors F_1 and F_2 come from the statistical standards tables and are taken from Table B.1 of ISO 13528 Standard – *Factors F_1 and F_2 for use in testing for sufficient homogeneity,* S_w is the standard deviation inside the sample or the laboratory, and $\sigma_{allow}^2 = (0{,}3\sigma_{pt})^2$.

To confirm the stability of the sample during the stablished analysis period, the samples are analysed at 0-time and final time. The 0-time match with the start of the round and with the homogeneity study, so the homogeneity results are taken as 0-time stability results. The final time stability study is carried out on the deadline established for carrying out the test. Three samples in duplicate are analysed and the results are studied following the criterion stablished on ISO 13528 Standard: the difference in absolute value between the average obtained at 0-time and the average obtained at final time must be less than or equal to 0,3 times the "target" deviation (σ_{pt}).

If this condition is not met, the criterion described at point B.5.2, section, from ISO 13528 Standard is verified - *It is likely that the intermediate precision of the measurement methods (or the uncertainty of measurement of the item) contributed to the inability to meet the criterion, then one of the following options should be taken – c) expand de criterion for acceptance by adding the uncertainty of the difference to* σ_{pt}:

$$|\bar{X}_{T0} - \bar{X}_{TF}| \leq 0{,}3\sigma_{pt} + 2\sqrt{\left(\mu_{\bar{X}_{T0}}{}^2\right) + \left(\mu_{\bar{X}_{TF}}{}^2\right)}$$

Where $\bar{X}_{T0}$ is the assigned value for 0-time stability, $\bar{X}_{TF}$ is the assigned value for final time stability and the uncertainties values $\mu_{\bar{X}_{T0}}{}^2$ and $\mu_{\bar{X}_{TF}}{}^2$ are calculated from the standard deviation of the replicates.

1.4. Statistical study.

The assigned value or reference value (**X_t**) for the analysed determinations in the QUALINOVA programs is stablished from the robust mean of the set of the results submitted by the participant laboratories (**$\bar{x}^*$**).

The robust statistical study is performed with the informatic application Robust Statistics Toolkit, from the Royal Society of Chemistry.

In order to minimise the effects caused in the robust statistics by outliers, values outside the range of the median value ± 50% are not taken into account. Such values are highlighted in the blue boxes.
In addition, results that have not been expressed according to the criteria set out in the instructions are eliminated:

- Results expressed ambiguously or with abbreviations.
- Results expressed in exponential form.
- Results expressed as Detected or Not Detected in case of parameters for which a quantitative result is requested.
- Results expressed numerically in case of parameters for which a qualitative result is requested.
- False Negative results, as they have been expressed as 0 or Not Detected when that micro-organism had been inoculated.

- False Positive results, expressed quantitatively or as Detected when this micro-organism had not been inoculated.
- Boxes with horizontal or oblique lines to express negative results or that the analyses have not been performed.

In cases where the participant indicates a value in the format 'below' (< Y), where Y is the laboratory's limit of quantification for that determination, or as 'greater than' (> Y), they shall not be considered for the statistical study and their performance shall not be evaluated. **ISO 13528:2022, point 5.5.3.4 and Annex E.1.**

In microbiological analyses, a logarithmic conversion ($\log_{10}$) of the results is performed, which allows to obtain a Gaussian (normal) distribution and to perform all statistical calculations in logarithmic format.

When a robust statistical study is performed, the formula for the calculation of the uncertainty of the assigned value is as follows:

$$\mu_x = \frac{1{,}25 \cdot S^*}{\sqrt{n}}$$ **(Ref.: ISO 13528:2022, point 7.7.7)**

where n is the number of participants in the round and S* is the robust standard deviation.

The evaluation of the effectiveness of each participant is done using the Z-score statistic, which is a measure of the deviations of the results from the assigned value (Xt). The Z-score is calculated as:

$$Z - score = \frac{(X_i - X_t)}{\sigma_{pt}}$$ **(Ref.: ISO 13528:2022, point 9.4.1)**

If $\mu_x > 0{,}3 \cdot \sigma_{pt}$, then the value calculated to assess the performance of each laboratory will not be Z-score, but Z'-score, which is calculated as follows:

$$Z' - score = \frac{(X_i - X_t)}{\sqrt{\sigma_{pt}^2 + \mu_X^2}}$$ **(Ref.: ISO 13528:2022, point 9.5.1)**

where σ_{pt} is the 'target' deviation, which estimates the appropriate dispersion of the results within each parameter, and μ_x is the uncertainty of the assigned value.

NOTE: The use of z'-score in exercises with the assigned value calculated by consensus may lead to an underestimation of the assigned value by approximately 10%, although its use is accepted (ISO 13528:2022, point 9.5.1.).

The acceptance criteria for **Z-score** and **Z'-score** for the evaluation of the effectiveness of participants are as follows:

Satisfactory	$\|z\| \leq 2$
Questionable	$2 < \|z\| < 3$
Insatisfactory	$\|z\| \geq 3$

To check for possible test multimodalities, skewness or other circumstances leading to a non-normal distribution, a Kernel density estimate is calculated using as a value:

$$h = \sigma_{pt} \cdot 0{,}75$$

In the event of an anomalous distribution, the possible causes of the anomaly shall be assessed. If no clear reasons for these anomalies are found, the assigned value shall be obtained by a resampling or bootstrap technique.

The participating laboratory is recommended to evaluate the results obtained in the proficiency test taking into account robust mean, robust standard deviation, target deviation, z-score data and graphical representations.

Should you require any further explanation for the understanding of the study conducted for the proficiency evaluation of the participating laboratories, please contact us at the following e-mail address: qualinova@labnovasl.com.

1.5. Results presentation.

For each parameter requested in the round, the results are presented according to the following scheme:

PARAMETER NAME

Table 1: Raw data of the participants
TABLE WITH THE RAW DATA OF THE PARTICIPANTS

Table 2: Preliminary elimination of results
TABLE WITH THE DATA ELIMINATED BEFORE STATISTICAL ANALYSIS
(**Table 3:** elimination criteria followed for each of the eliminated participants)

Statistical results:

Assigned value (X_t)	
Robust standard deviation (S^*)	
Number of participants (n)	
Uncertainty of the assigned value (μ_x)	
Standard deviation for proficiency assessment (σ_{pt})	

Statistic to be used to evaluate the performance: Z-Score or Z'-Score following the critera $0,3 \cdot \sigma_{pt} > \mu_x$

Table 4: Results of the performance evaluation.

Laboratory	Result	Units	Z-score or Z'-score	Method
Identification code of each laboratory	Parameter value for each laboratory	Units in which the result is expressed	Z o Z' of each laboratory	Used method

Value of Z or Z' score > |2|

Questionable and **unsatisfactory** results.
Percentage of **satisfactory** results.

HISTOGRAM OF Z/Z'-SCORE VALUES OF ALL LABORATORIES

KERNEL DENSITY OF THE PARAMETER
(If Bootstrap is performed, this section will be before the statistical results)

In the case of microbiological parameters, a column is added next to the result column for results in logarithmic units.

For qualitative parameters, no statistics are calculated for performance evaluation, so the presentation of results follows a different scheme:

PARAMETER NAME

Table 1: Raw data of the participants
TABLE WITH THE RAW DATA OF THE PARTICIPANTS

Table 2: Preliminary elimination of results
TABLE WITH THE DATA ELIMINATED BEFORE STATISTICAL ANALYSIS
(**Table 3:** elimination criteria followed for each of the eliminated participants)

Table 4: Results of the performance evaluation.

Laboratory	Result	Units	Method	Evaluation
Identification code of each laboratory	Parameter value for each laboratory	Units in which the result is expressed	Used method	Correct = ☑ o Incorrect = ☒

Micro-organism inoculated and target concentration attempted → Expected qualitative result..

Unsatisfactory results based on inoculation

Number of **satisfactory results.**
Number of **unsatisfactory results.**
Percentage of satisfactory results.

COMMENTARY ON THE RESULTS OF THE PARAMETER

GRAPHICAL REPRESENTATION OF THE PERCENTAGES OF RESULTS

2. ROUND DETAILS

2.1. Characteristics of the item.

bottles, vials and …. were provided.

- Vial X: inoculated microorganisms
- Bottle X: quantity and parameters

Samples are prepared with the aim of obtaining as natural a sample as possible.

The samples of the Qualinova ENVIRONMENT/FOOD Program, for physico-chemical analysis, are real samples and by their very nature the sample did not need to be doped with additional reagents to reach the desired concentrations in the proficiency test.

The shipment was carried out through a transport agency, in a XXXXXX (shipping container) to protect against shocks. The instruction sheet was sent via e-mail, specifying the dates set for the analysis and the deadline for sending the results, and the method to be followed for the correct preparation of the sample.

Date of shipment	xx-xx-xxxx
Date of analysis	xx-xx-xxxx
Closing date of the round	xx-xx-xxxx
Date report issued	xx-xx-xxxx
Number of participants convened	xxx

2.2. Preparation method for the item

The method specified in the instruction sheet sent via email is followed.

2.3. Parameters and σ_{pt} values.

The following table lists the parameters to be analysed, the microorganisms inoculated for each of them and the "target" deviation values (σ_{pt}) that have been considered (*fit for purpose)* and that have been obtained by using the criterion xxxxx.

VIAL	PARAMETER	INOCULATED MICROORGANISMS –CECT CODE	σ_{pt} Value
A	Total coliforms	*Escherichia Coli* CECT 434	0,25

2.4. Homogeneity and stability results.

The tests for homogeneity and stability studies were performed by external analytical service providers accredited according to ISO 17025.

The homogeneity and stability studies were in all cases satisfactory according to the criteria set out in ISO 17043.

3. RESULTS ANALYSIS

3.1. PARAMETER 1

4. COMMENTS AND RECOMMENDATIONS BASED ON RESULTS AND PERFORMANCE

END OF REPORT

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General report format in force, updated in July 2024.