PROFICIENCY TESTS
QUALINOVA
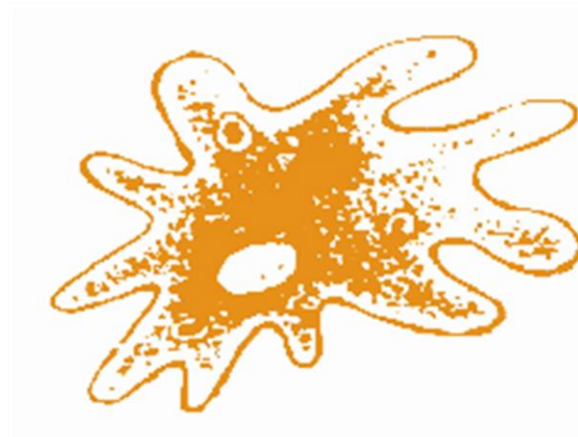
QUALINOVA XXXXXXX

CIRCUIT XXXXX

ROUND: XXXXX

Date of issue: XX-XX-XXXX

Review 0

This report has been drafted and approved by:

SIGNATURE– NAME

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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This report presents the results of the XX XXXXX round of the Proficiency Testing Programme: Qualinova XXXX in which it participated.

The analysis of results and the statistical treatment have been carried out on the data provided by the participants in computerised form, without any other transcription or manipulation of the data.

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

1. GENERAL ASPECTS

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

Some general aspects are commented below.

The organisation and development of 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.
- Specific ISO standards for the parameters and matrices to be analysed in each case.

Any activity related to the round preparation process can be outsourced to a company that follows the requirements of the proficiency testing provider, based on ISO 17043. The parameter analyses are carried out by a laboratory accredited under ISO 17025.

Each round is independent of each other, with no minimum number of rounds to sign up for. The general structure of each of the rounds that make up the programmes is as follows:

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

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▪ The organisation provides an instruction sheet via email, which is necessary to carry out the corresponding analyses. It is necessary to treat the sample according to the instructions on this sheet.

▪ In each exercise, all or only part of the proposed analyses can be carried out.

▪ Participating laboratories register the results obtained through the website www.labnovasl.com/qualinova, accessing the client portal with the user's name and password provided by the organisation.

▪ The values entered must be indicated with a dot as a thousand separator (.), and decimals must be separated by a comma (,). Microbiology results should be expressed in the requested units, but without 7XE03 or powers of 10. Figures should be whole numbers: 7,000, 1,000,000 etc. The number of significant figures will depend on the sensitivity of the method used.

▪ Each participant must also indicate the method used in each analysis in the corresponding box on the results submission form.

▪ Once all the results have been received, the Qualinova organisation carries out the statistical processing of the results and prepares the final report for this round, together with the personalised report for each participant.

▪ The participating laboratories receive the report of the results where the quality of their results is evaluated. Each laboratory is identified by a code number that is known only to the laboratory, thus ensuring the confidentiality of the results. This number is indicated in the personalised report that accompanies the general report to each participant.

▪ In the event that a participant wishes to submit a complaint about the evaluation of his/her performance in a round, he/she should contact the e-mail address qualinova@labnovasl.com, where his/her request will be assessed.

1.1. Definitions.

Proficiency testing provider: Organisation 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 two or more laboratories in accordance with predetermined conditions.

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

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Proficiency testing 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 ser of values which is inconsistent with other members of that set.

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

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

Standard deviation for proficiency assessment (σ_{pt}): Measure of dispersion used in the evaluation of results of *proficiency testing.*

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

Z-score: Standardized measure of performance, calculated using the participant result, *assigned value* and the *standard deviation for proficiency assessment.*

1.2. Standard deviation for proficiency assessment

According to ISO 13528, these are the possible approximations for the estimation of σ_{pt}:

A. <u>Expert perception</u>. The maximum permissible error of (σpt) can be set as the value that corresponds to the level of performance that a regulatory authority, accreditation body or the proficiency testing provider's technical experts consider to be reasonable for the participants.

B. From <u>experience gained</u> in previous rounds of a proficiency test.

C. <u>Use of a general model</u>, commonly used in the chemical field, is that 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 to calculate the standard deviation of reproducibility.

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

E. Use of the <u>participant's own data obtained</u> in the Proficiency Testing round itself. It should be noted that the use of the participants' own results may result in a criterion for performance assessment that may not be appropriate. The proficiency testing provider should always ensure that the (σpt) used is fit for purpose.

In the case of microbiological parameters, a combination of approaches A and B shall be followed, based on the experience of the PT provider's expert from previous years' rounds and on point 8.3.6.2 of UNE-EN ISO 22117: *'The calculation of the z-index uses an ideal standard deviation value (σpt). This ideal standard deviation defines the range of acceptable variation between laboratories for a particular test, with σpt values of 0.35 or 0.25 being commonly used in microbiological AE schemes'*.

In the case of physic-chemical parameters, whenever a regulation or legislation exists with data on tolerances, uncertainties or precision, these data shall be used to set the σpt values. If no legislation exists, the expert of the proficiency testing provider shall be consulted and historical data shall be reviewed. As a next option the general Horwitz model modified with Thompson shall be used.

1.3. Homogeneity and stability

The samples have been subjected to a homogeneity and stability study, according to the protocol established in the ISO 17043 and ISO 13528 Standards, keeping the samples in the same conditions in which they will be received at the destination laboratories.

To carry out the homogeneity study, 10 samples are taken at random from all the prepared vials and two portions of each one are analysed under repeatability conditions. The homogeneity criterion established in ISO 13528, whereby the standard deviation between samples (Ss) must be less than or equal to 0,3 times the 'target' deviation (σ$_{pt}$), is checked for compliance.

If this condition is not fulfilled, the criterion indicated in section B.2.3 of ISO 13528 is checked – *It may be useful to expand the criterion to allow for the actual sampling error and repeatability in the homogeneity check* (it may be useful to extend the criterion to take into account the actual sampling error and repeatability in the homogeneity check). The sample is accepted as homogeneous as long as it complies with: $s_s < \sqrt{c}$, donde $c = F_1 \sigma_{allow}^2 + F_2 s_w^2$ y $\sigma_{allow}^2 = (0{,}3\sigma_{pt})^2$.

To confirm the stability of the sample during the established analysis period, samples are analysed at time 0 and end time. Time 0 coincides with the start of the round and with the homogeneity study, therefore the homogeneity results are taken as stability results at time 0. The end time stability study is performed at the deadline for the submission of results. Three samples are analysed in duplicate and the results are studied according to the criteria established in ISO 13528: the difference in absolute value

between the mean obtained at time 0 and the mean obtained at end 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 in point B.5.2, paragraph c, of ISO 13528 is checked - *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}* (It is likely that the intermediate precision of the measurement methods (or the measurement uncertainty of the item) has contributed to the failure to meet the criterion, then one of the following options should be taken: c) extend the acceptance criterion by adding the uncertainty of the difference to σ_{pt}):

$$| \bar{x}_{T0} - \bar{x}_{TF}| \le 0.3\sigma_{pt} + 2 \sqrt{(u^2(\bar{x}_{T0})+ u^2(\bar{x}_{TF}))}$$

where the values of the uncertainties $u^2(\bar{x}_{T0})$ y $u^2(\bar{x}_{TF})$ are calculated from the standard deviation of the replicates.

These tests have been carried out by one or more external service provider(s) validated and approved by the proficiency testing provider, subcontracted for this purpose by the Qualinova organisation.

1.4. Statistical study

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

The robust statistical study is carried out with the Robust Statistics Toolkit software application of 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, values that do not comply with the requested format are removed:

- 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.

- Results expressed as 0 or Not Detected when that micro-organism was inoculated.

- Results expressed quantitatively or as Detected when this micro-organism was not inoculated.

- Spaces or empty cells because the analysis was not performed. Boxes with horizontal or oblique lines to express negative results or that tests have not been performed.

In cases where the participant indicates a value in the format 'less than' (< Y), where Y is the laboratory limit of quantification for that determination, the approximation XXXXXXXXXXXXXXXXXXXXXXXXXXXXXX specified in ISO 13528:2022, point 5.5.3.4 and Annex E.1 has been followed.

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

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

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 (X_t). The Z-score is calculated as:

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

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:

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

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	$|z| \leq 2$
Doubtful	$2 < |z| < 3$

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Unsatisfactory	|z| ≥ 3

To check for possible test multimodalities, asymmetries or other circumstances causing a non-normal distribution, a Kernel density estimate is calculated using **h = $\sigma_{pt} \cdot$ 0,75.**

If an anomalous distribution is found, the possible causes of the anomalous distribution shall be assessed. If no clear reasons for these anomalies are found, the assigned value will 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. Presentation of results

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

NAME OF THE PARAMETER

Chart 1: Raw data of the participants
CHART WITH THE RAW DATA OF THE PARTICIPANTS

Chart 2: Prior elimination of results
CHART WITH THE RESULTS ELIMINATED PRIOR TO THE STATISTICAL STUDY
(**Chart 3:** elimination criteria that has been followed for each of the eliminated participants and the percentage of data eliminated.)

Chart 4: Results of the effectiveness evaluation.

Laboratory	Result	Unit	Effectiveness evaluation (Z-score o 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 score > |2|

X_t Valor asignado por consenso	H15 Mean	
S* Desviación estándar robusta	H15 Std Dev	
n Número de resultados participantes en el estudio		
μ_x Incertidumbre del valor asignado		
σ_{pt} Desviación diana		
0,3·σ_{pt}	**Si μ_x > 0,3·σ_{pt}, se utilizará Z'-score**	

Statistic to be used to assess efficacy: Z-Score or Z'-Score according to criterion 0.3 - σ_{pt} > μ_x
Doubtful and unsatisfactory results.
Percentage of satisfactory results.

COMMENTARY ON THE RESULTS OF THE PARAMETER

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

KERNEL ESTIMATE OF THE PARAMETER

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

For qualitative parameters no statistics are calculated for the evaluation of efficacy and therefore the presentation of the results follows a different scheme:

NOMBRE DEL PARÁMETRO

Chart 1: Raw data of the participants CHART WITH THE RAW DATA OF THE PARTICIPANTS

Chart 2: Prior elimination of results CHART WITH THE RESULTS ELIMINATED PRIOR TO THE STATISTICAL STUDY (**Chart 3:** elimination criteria that has been followed for each of the eliminated participants and the percentage of data eliminated.)

Chart 4: Results of the effectiveness evaluation.

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

Inoculated micro-organism and inoculated concentration. ☐ Expected qualitative result.

Unsatisfactory results as inoculated.

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.   DETAILS OF THE ROUND

2.1. Item characteristics

A homogeneous and stable material is provided in the form of x vials containing freeze-dried micro-organisms together with a sterile matrix of xxxx.
    - Vial A:
    - Vial ...
    X vials, vial A quantity... and vial B....

The samples are prepared with the aim of obtaining a sample as real as possible to those found in nature or in our daily life.

The samples of the Qualinova ENVIRONMENT/FOOD Programme, for physic-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 has been made via freight forwarder, in a xxxxx (shipping container) as protection 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 dispatch	xx-xx-xxxx
Date of analysis	xx-xx-xxxx
Deadline for the round	xx-xx-xxxx
Date of issuance of the report	xx-xx-xxxx
Number of participants convened	XXX

2.2. Test item preparation method

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

2.3. Parameters and values of σ_{pt}.

The following table lists the parameters to be analysed, the expected qualitative results according to what is inoculated/present in the sample and the 'target' deviation values (σ_{pt}) that have been considered *fit-for-purpose* and that have been obtained using the criterion xxxxx.

PARAMETER	INOCULATED MICROORGANISM	σ_{pt} VALUE

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Total coliforms	*Escherichia Coli*	0,25

2.4. Homogeneity and stability results.

Homogeneity and stability studies carried out by external service providers according to the criteria of ISO 17043 have given satisfactory results for the use of the sample as a proficiency test item.

3.   ANALYSIS OF THE RESULTS

4.   COMMENTS AND RECOMMENDATIONS BASED ON RESULTS AND PERFORMANCE

END OF REPORT

QUALINOVA XXXXXXX
SCHEME XXXXXXXX
ROUND XXXXXX
Review 0
DATE OF ISSUE XXXXXX

DRAFTED BY	REVISED BY
Date:	Date: