



TestQual, S.L.
(Proficiency Testing Schemes)

*Pol. Industrial Oeste
Av. Principal, Parcela 21/1
CP 30169, San Ginés, Murcia
Telephone: 868 949 486 / 676 367 555*

TestQual 121 PROTOCOL
Acrylamide in Potato chips

1. INTRODUCTION

This document describes the **protocol** of the **TestQual 121** Proficiency Test (P.T.), which belongs to the analysis of **Acrylamide** in **Potato chips**.

TestQual, S.L. is committed to maintain the confidentiality of all laboratories from the beginning of the proficiency test and after it.

2. OBJECTIVE

The objective of the **TestQual 121** Proficiency Test is to evaluate the quality and accuracy of the results sent by the participating laboratories. Because of this, proficiency testing is an essential element of laboratory quality assurance. It helps to control and detect errors in their results or methods of analysis.

3. CALENDAR

The following table shows the schedule for this proficiency test:

Date	Activity	Carried out by
<i>08/Nov/19</i>	<i>Final date to receive applications</i>	<i>Participants</i>
<i>12/Nov/19</i>	<i>Sample delivery</i>	<i>TestQual</i>
<i>06/Dec/19</i>	<i>Final date to receive results</i>	<i>Participants</i>
<i>Week 50 to 52 December</i>	<i>Final report</i>	<i>TestQual</i>

The dates of this calendar might vary slightly according to the development of the P.T. during the year. However, any modification in the dates will be announced in advance on our website www.testqual.com.

The **coordinators** of this proficiency test will be Jose Pedro Navarro and María Ángeles Garrido. Any question regarding the development of the P.T. can be consulted by email jpnavarro@testqual.com or magarrido@testqual.com

4. PARTICIPATION REQUEST AND WEBPAGE

WEBPAGE

TestQual webpage and all of its documents are available in both English and Spanish.

NEW CLIENT

If your laboratory has not participated before in one of our proficiency tests you will have to register on our website using the REGISTER form.

Once you have completed and sent the form you as soon as possible the website administrator will activate your account. If some more information is needed to complete your registration one

of the PT coordinators will get in contact with you through the contact data you introduced during your registration.

If you have any doubt or if we can help you with anything you can always contact any of our PT coordinators, you will find all the contact data in the [Contact tab](#) from our website.

For those laboratories that want to have more than one contact per account or wants more than one laboratories per account will have to contact one of the PT coordinators or through our website to be instructed how to proceed.

APPLICATION FOR THE PROFICIENCY TEST

To participate in this proficiency test the application is needed to be done through the website. You can send your application by entering our website and go to the [Proficiency tests tab](#), if you are interested in one of our PTs, by clicking on the name of the PT or the shopping cart you will be able to see general information about that PT, in that page is also available to check and download the present document (the protocol) and at the bottom of the page will be a link to start the [APPLICATION FORM](#), all inscriptions must be done before the scheduled date in the calendar.

Once the application has been sent, as soon as possible, it will be checked by the website administrator and you will be sent an email with the participation code. This code will be just known by the organizer and the laboratory, and will be kept confidential at all times.

Just one application per exercise can be sent by each laboratory, being not allowed for a laboratory to participate with two different codes.

Laboratories with two or more laboratories in its network can participate with all of their laboratories in the same PT. Different applications are needed for each laboratory. In case of doubt you can contact the PT coordinator for guidance.

The applications of the laboratories will be studied and accepted in base of the quantification limits of the analytes of the P.T

TestQual can anticipate that the number of participants of this P.T. will be around 11, being 11 the minimum participants for this proficiency test to take place.

5. TEST MATERIAL

TestQual 121 scheme is a proficiency test based in the analysis of **Acrylamide** in **Potato chips**. The material is bought to a specialised company in Murcia and analysed by a subcontracted laboratory that holds the standard UNE-EN ISO/IEC 17025 into force.

The material is crushed and poured into a homogenizer to ensure complete homogeneity, then it is packed in a special plastic bag that is thermally sealed in vacuum, then is stored in a cold, dark place until distribution.

For [homogeneity](#) assessment purpose, ten of the prepared samples are analysed in duplicate by TestQual's collaborator laboratory under repeatability conditions.

For stability assessment purpose, three samples are analysed, in duplicate, before, during and at the end (once all laboratories have sent their results) of the proficiency test.

6. SAMPLE SHIPMENT

The shipment of the test materials will take place on the date shown in the calendar, to the address provided by each laboratory in the application. Specific delivery dates may change from the scheduled dates of the calendar, but any change will be announced both on the website and by email to the participants.

About **100 g** of test material will be sent by courier service (MRW, DHL or TNT, depending on the destination). The material will be sent in padded envelope that ensures proper conditions of arrival of the sample. The transit will be 1, 2 or 3 days, depending on the location of the receiving laboratory.

A second sample may be requested within two days from the reception of the first one by any participant who finds the package or the sample damaged.

7. CONCENTRATION RANGES, SIGMA OBJECTIVE AND ANALYTES

In this proficiency test, the acrylamide will be found above **10µg/Kg**.

The **sigma objective ($\hat{\sigma}$)** which works in this scheme will be the **20% of the assigned value**. This value was chosen based on the Thomson-Horwitz equation for concentrations higher than 120µg/Kg.

The result of the laboratory should be expressed as the content of **Acrylamide** in µg/Kg.

8. RESULTS EXPRESSION

Each participant must analyse the sample received according to their routine procedures, and fill up the RESULTS form of its private area of the website www.testqual.com with only one value.

The results should be expressed in **µg/Kg**. The number of significant figures and the units are shown as they are sent by the laboratories.

The method used for the analysis should be sent when filling up the results form.

The organizer should get the results before the deadline of the scheme.

9. STATISTICAL EVALUATION

TestQual will develop the following statistical evaluation:

TestQual considers as an **extreme outlier** any data which differs more than **50 %** of the average of all results reported by the laboratories, according to the Harmonize Protocol of the IUPAC. These extreme values will not be included in the calculation of the assigned value.

Once received all the results, TestQual evaluates the unimodality of all the values with a histogram built with the Kernel densities, being explained in the final report which is the followed procedure in case there is more than one distribution.

The **assigned value (X)** is determined using the robust average of the results considered valid for statistical computing (after eliminating the extreme outliers), according to the standard ISO13528 up to date.

The **standard uncertainty (u_x)** is calculated using robust statistics from the following formula:

$$u_x = 1,25 \cdot (s^*/\sqrt{p})$$

Being s^* the robust standard deviation of the data and p the number of results considered.

The following condition must be fulfilled in order to discard the contribution of the uncertainty:

$$u_x \leq 0,3 \hat{\sigma}$$

In case this condition is not fulfilled, the participants of the scheme will be informed, and the uncertainty will have to be taking into account for the assigned value assessment or the evaluation will be given with informative purposes.

The **standard deviation for proficiency assessment**, also named **target standard deviation, ($\hat{\sigma}$)**, comes from this formula:

$$\hat{\sigma} = b_i \cdot X$$

Being $b_i = \%_{DSRA} / 100$, and $\%_{DSRA}$ is the assigned relative standard deviation.

In this case, the assigned relative standard deviation is **30 %**. This value is fixed previously by the organizer based in the experience of TestQual organizing similar proficiency tests.

Proficiency assessment (z-score): This parameter shows the competence and accuracy of the laboratory. It is calculated using the following formula:

$$z = (x_i - X) / \hat{\sigma}$$

Where x_i is the value reported by the laboratories, X is the assigned value, and $\hat{\sigma}$ is the target standard deviation for each analyte.

The criteria for defining the z-score values are:

	$ z \leq 2$	<i>Satisfactory</i>
$2 <$	$ z \leq 3$	<i>Questionable</i>
	$ z > 3$	<i>Unsatisfactory</i>

False negatives: Any analyte not reported in the results that were in the sample above the limit of quantification previously established to the proficiency test established by the organization (**10 µg/Kg**). TestQual assigns to all false negatives a result equal to half the laboratory limit of quantitation (LOQ/2).

False positives: Those analytes reported in the results, which were not present in the test material, are reported by the participant at concentrations higher than the limit of quantification of the P.T. (**10 µg/Kg**).

Testing for sufficient homogeneity:

Once the samples were prepared, ten of them were chosen at random and sent to be analysed by TestQual's collaborator laboratory. Once received the results, a statistical evaluation was performed, according to the IUPAC Harmonic Protocol.

The acceptance criterion to ensure that the randomly chosen samples were homogeneous was that the square of the estimated sampling standard deviation is below the critical value for accepting proper homogeneity:

$$S_{sam}^2 < c$$

In the first place to check the criterion, S_{sam}^2 which is the estimated sampling standard deviation, is calculated from:

$$S_{sam} = \left(\frac{V_s}{2} - S_{an} \right)$$

Firstly V_s is the variance of the sums S_i :

$$V_s = \sum \frac{(S_i - \bar{S})^2}{m - 1}$$

Where S_i can be obtained from the addition of each duplicate result from the homogeneity; \bar{S} is the mean of all S_i and m is the number of samples (10 samples).

And secondly S_{an}^2 , which is the experimental estimate of analytical standard deviation, is obtained following formula:

$$S_{an}^2 = \frac{\sum D_i}{2m}$$

where D_i is the result of the subtraction of each pair of replicates from the homogeneity and m is the number of samples.

Lastly to calculate the critical value c was obtained from:

$$c = F_1 \cdot \sigma_{all}^2 + F_2 \cdot S_{an}^2$$

Being F_1 and F_2 constants with values equal to 1.88 and 1.01 respectively for 10 samples. S_{an}^2 has already been calculated and σ_{all}^2 is obtained from:

$$\sigma_{all}^2 = (0.3 \cdot \hat{\sigma})^2$$

where $\hat{\sigma}$ is the target standard deviation, which is calculated with the formula:

$$\hat{\sigma} = 0.2 \cdot \bar{X}$$

Being \bar{X} , the mean of the 20 values from the homogeneity.

Testing for sufficient stability:

Three samples will be analysed, in duplicate, before, during and at the end (once all laboratories have sent the results) of the proficiency test. The acceptance criteria to ensure the samples have been stable during the whole P.T. are the following:

$$|(X_{t1} - X_{t2}) / X_{t1}| \cdot 100 \leq 10\%$$

$$|(X_{t1} - X_{t3}) / X_{t1}| \cdot 100 \leq 10\%$$

Being $|(X_{t1} - X_{tn}) / X_{t1}|$ the difference between the average of the samples analysed before, during and at the end of the proficiency test.

10. EVALUATION REPORT

Once received and statistically evaluated all of the participating laboratories results, TestQual will send a final report that summarizes the participation of each laboratory.

This final report will sent to the laboratories via e-mail in PDF format, and it can also be downloaded from the client area of each participant on www.testqual.com. Laboratories may request TestQual to send them the reports in paper by mail as well.

If any participant wants to appeal against the assessment program performance, a written appellation must be sent by email to jpnavarro@testqual.com or magarrido@testqual.com, explaining their reasons for it.

11. REFERENCES

TestQual Proficiency Testing Schemes are based on the following standards:

UNE-EN ISO/IEC 17043, first edition 2010-02-01. Conformity assessment- General requirements for proficiency testing.

ISO13528:2015, second edition 2015-08-01. Statistical methods for use in proficiency testing by interlaboratory comparison.

THE INTERNATIONAL HARMONIZED PROTOCOL FOR THE PROFICIENCY TESTING OF ANALYTICAL CHEMISTRY LABORATORIES

~~*SANTE/11945/2015, 30 November 1 December 2015 rev.0 Guidance document on analytical quality control and method validation procedures for pesticides residues analysis in food and feed.*~~