



TESTQUAL

PROFICIENCY TESTING SCHEMES

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(Proficiency Testing Schemes)

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TestQual 162 PROTOCOL

Pesticides in Paprika

Summary of changes

Changes marked with *blue* or **highlighted blue**.

Rev01 →

- Acceptance deadline of applications updated.
- Text added regarding LOQs and applications acceptance.
- More info added about subcontracting done.
- If z'-score is issued, it will be accredited and the difference against z-score will be included in the report.
- Other minor changes

Rev02 → Calendar updated, changes in statistical design: new alternative option assigned value from spiking, homogeneity test.

Rev03 → Calendar updated.

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0. GLOSARY AND ABBREVIATIONS

Text	Abbreviation
TestQual	TQ
Proficiency test	PT / P.T.
Limit Of Quantification	LOQ
Not Analysed	NA

1. INTRODUCTION

This document describes the **protocol** of the **TestQual 162** Proficiency Test (P.T.), belonging to the analysis of **Pesticides** in **Paprika**.

In the present document is detailed how to start working with TestQual, send your application to participate, the statistic that will be applied and information about the evaluation report.

TestQual, S.L. is committed to maintaining confidentiality with the information of each laboratory from the beginning of the proficiency test.

2. OBJECTIVE

The objective of the **TestQual 162** 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 will help to control and detect errors in their results or methods of analysis.

3. CALENDAR

The following table shows the program for this proficiency test:

Date	Activity	Carried out by
-	Deadline to reserve PT sample. Ensured reviewing* of application.	Participants
Open until samples are sent	Deadline to send application (acceptance depends on availability)	Participants
Week 30 (25/Jul & 26/Jul/22)	Sample delivery	TestQual
Week 36 (09/Sep/22)	Final date to submit results	Participants
Week 38 (23/Sep/22)	Final report (Email and/or client area)	TestQual

*Participants are requested in the application to submit their LOQ/LOQs, for PTs with multiple possible analytes, if participants analyse above a certain percentage (as described in our internal procedure) of present/planned analytes the participation is accepted, a laboratory code is granted and sent by email to confirm the participation acceptance to the user of the TestQual's account.

The dates of this calendar might be slightly changed according to the development of the proficiency test during the year. However, any change would be notified to all participants announcing it on our website www.TestQual.com.

The **coordinator** of this proficiency test will be Jose Pedro Navarro. Vicente. Any question regarding the development of the proficiency test, the application status or any other query can be consulted by email to jpnavarro@testqual.com.

4. REGISTER AND PARTICIPATION REQUEST (APPLICATION FORM)

NEW CLIENT

If your laboratory has not participated before in one of our proficiency tests you will have to register on the [REGISTER](#) form.

Once you have completed and sent the form you will have to wait until the activation of the account from the website administrator. If some more information is needed someone from our team will get in contact with you through the phone or email you used during your registration.

You can find our contact data at the end of this protocol.

For those laboratories that require more than one contact per account or are in a situation not contemplated in this protocol will have to contact the organizer using the Contact tab to be instructed how to proceed.

APPLICATION FOR THE PROFICIENCY TEST

To participate in this proficiency test or be updated via email of any changes regarding this proficiency test is required to have a laboratory code. To get your laboratory code is needed to apply through the website or have it arranged by the coordinator at least 24h before the shipping of the samples.

In the Proficiency Tests Tab on our website, you will have to select the proficiency test you want to participate, by clicking its name or the shopping cart you will enter the page with general information and a summary of that proficiency test, there you can find the present document (the protocol) and the button to start the application.

If you did not log in before you will be requested to do so and then the website will require you to submit your Limit Of Quantification (LOQ) for the parameters you will study. The compounds left as NA (NOT ANALYSED) will NOT appear in the Results form and therefore will not be able to send results for those parameters through the website.

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 only by the organizer and the participant and will be always kept confidential, even after the proficiency test finishes.

You can check on the dashboard of your client area if an application you sent has been accepted or is still pending.

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

The applications of the laboratories will be studied and accepted in base of the quantification limits of the analytes of the proficiency test and if the logistics allow the sample shipping without risk of deterioration.

According to the experience, TestQual can anticipate that the number of participants of this proficiency test will be between 5-15 participants.

5. PREPARATION AND QUALITY CONTROLS

TestQual 162 scheme is a proficiency test based in the analysis of **Pesticides in Paprika** that has been spiked with **standards**.

The material will be bought in an specialized shop in Spain and analysed by a subcontracted laboratory that holds the standard UNE-EN ISO/IEC 17025 into force.

The material is spiked with a solution with the analytes of the proficiency test, homogenised at a controlled temperature and stored in a dry fresh place without light until further distribution to participants.

The distribution of samples was subcontracted to a courier previously homologated by TestQual. The main criteria being the courier's delivery time to ensure the receival of the sample is correct in the participant's facilities.

In addition to this, TestQual stablished other characteristics important for a courier like shipping management (tracking, notifications, exceptions), and ensuring the delivery conditions are proper (low breakage/lost ratio, keeping of cold chain, required documentation), always checking and evaluating they are complying with TestQual's requirements.

Before the samples are distributed, for the assessment of the homogeneity of the lot of samples prepared, ten samples from the lot will be selected randomly and analysed in duplicate by TestQual's collaborator laboratory under repeatability conditions. If the mean concentration obtained from this study is not within the planned range, the participants will be informed and a new distribution day might be scheduled if another spiking is deemed as necessary.

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

The quality controls subcontracted by TestQual, including verification of adequacy of the matrix, homogeneity/stability quality controls and any other analytical study required by TestQual will be subcontracted to an accredited laboratory in ISO/IEC 17025 into force.

In the evaluation report will be included the conclusions and if applies, any comment regarding homogeneity &/or stability. Additionally, the results of these tests and the spiking evaluation is available to all participants of this proficiency test upon request.

6. TEST MATERIAL AND 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 can change from the scheduled dates of the calendar, but all changes will be announced both in the website and by mail to the registered laboratories.

This PT will consist in a single round in which will be sent a sample of approximately **50-100 g** of test material. The samples will be sent by courier service (MRW, FedEx, DHL or TNT, depending on the destination).

The material will be sent with materials and package that ensures the samples arrives correctly. The transit will be 1, 2 or 3 days to the destination country, depending on the location of the receiving laboratory.

The shipping conditions for this PT are:

ROOM TEMPERATURE, The material will be sent in a vacuum packed, heat sealed plastic bag in dark conditions (opaque and padded envelope).

The shipping costs are not included in the price displayed on the website, which can only be seen if you are registered and logged in. To get an approximation of the shipping costs you can get your quotation by using the contact data at the end of this protocol.

A second test material can be requested date if necessary. If the package and/or the sample arrived damaged, defective, or not valid the participating laboratory will have to notify of this to the coordinator before two working days to get another sample.

Before the shipment, TestQual will send the instructions for storage and analysis via email and confirm the distribution date. You can request a paper copy to be attached to the package and/or TestQual might decide to include it in addition to have it sent by email.

From TestQual we encourage our participants to read it carefully and follow its instructions, as it can help to correctly conserve the sample and increase the reproducibility of the analysis.

You can request a digital copy of this document by letting us know through any communication channel.

7. CONCENTRATION RANGES, SIGMA OBJECTIVE AND ANALYTES

For this proficiency tests the range of concentration for the target analytes might be between **10** and **200 µg/Kg approximately**. This range is not absolute and depending on the various factors some or no analyte might be within this range. This range is given to provide participants a sense of the concentration ranges we usually work and expect in these kind of proficiency tests (combination of analyte/matrix).

The maximum concentration can vary depending on the MRL of the analytes in the matrix.

The **sigma objective** ($\hat{\sigma}$) which works in this scheme can be checked on section 9 of this protocol.

The **possible pesticides** in the Paprika are from the list below:

2-Phenylphenol	Chlorpyrifos	Dioxathion	Flutolanil
3,5-Dichloroaniline	Chlorpyrifos Methyl	Diphenylamine	Flutriafol
3-Hydroxy-carbofuran	Chlorthion	Dipropetryn	Folpet
4,4-	Chlorthiophos	Disulfoton	Fonofos
Dichlorobenzophenone	Cyanazine	Ditalimfos	Formothion
Abamectin	Cyazofamid	Diuron	Phosalone
Acephate	Cyfluthrin	Dodine	Phosphamidon
Acetamidrid	Cymoxanil	Emamectin benzoate	Phosmet
Acetochlor	Cypermethrin	B1a	Furalaxyl
Aclonifen	Cyproconazole	Endosulfan-alpha	Furathiocarb
Acrinathrin	Clethodim	Endosulfan-beta	HCH-Alpha
Alachlor	Clofentezine	Endosulfan-sulfate	HCH-Beta
Aldicarb	Clomazone	Endrin	HCH-Delta
Aldicarb sulfone	Cloquintocet-mexyl	EPN	HCH-Gamma (lindane)
Aldicarb sulfoxide	Chlorfenson	Epoxiconazole	Heptachlor
Aldrin	Chlorotoluron	Etaconazole	Heptachlor-epoxide
Antraquinone	Chloroxuron	Ethion	Heptenophos
Atrazine	Chlorpropham	Ethoprophos	Hexachlorobenzene
Azaconazole	Chlorsulfuron	Etoxazole	Hexaconazole
Azinphos-ethyl	Chlorthal-dimethyl	Ethiofencarb	Hexaflumuron
Azinphos-methyl	Clothianidin	Ethiofencarb -sulfone	Hexazinone
Azoxystrobin	Coumaphos	Ethiofencarb -	Hexythiazox
Benalaxyl	Kresoxim-methyl	sulfoxide	Imazalil
Bendiocarb	Crimidine	Etofenprox	Imazamethabenz-
Benfluralin	Cyanofenphos	Ethofumesate	methyl
Benfuresate	Cyanophos	Etrimfos	Imidacloprid
Bentazone	Cycloxydim	Famoxadone	Indoxacarb
Bifenthrin	Cyprodinil	Famphur (Famophos)	Iprobenfos
Bitertanol	Deltamethrin	Fenarimol	Iprodione
Boscalid	Demeton-S-methyl	Fenazaquin	Iprovalicarb
Brodifacoum	Demeton-S-methyl	Fenbuconazole	Isazofos
Bromacil	sulfone	Fenbutatin oxide	Isocarbophos
Bromocyclen	Desmetryn	Fenchlorphos	Isofenphos
Bromophos-ethyl	Dialifos	Fenhexamid	Isofenphos-methyl
Bromophos	Diazinon	Fenitrothion	Isoproturon
Bromopropylate	Dicapthon	Fenoxycarb	Lambda-Cyhalothrin
Bromuconazole	Dichlofenthion	Fenpropathrin	Lenacil
Bupirimate	Dichlormid	Fenpropimorph	Leptophos
Buprofezin	Dichlobenil	Fenpyroximate	Linuron
Butafenacil	Diclobutrazol	Fensulfothion	Lufenuron
Butamifos	Dichlofluamid	Fenthion	Malaoxon
Butoxycarboxim	Diclofop-methyl	Phenthoate	Malathion
Butralin	Dicloran	Fenuron	Mecarbam
Buturon	Dicrotophos	Fenvalerate	Mefenpyr-diethyl
Cadusafos	Dieldrin	Fipronil	Mepanipyrim
Captan	Diethofencarb	Flonicamid	Mepronil
Carbaryl	Difenoconazole	Fluazifop-P-butyl	Metalaxyl
Carbendazim	Difenoxuron	Fluchloralin	Metamitron
Carbophenothion	Diflubenzuron	Flucythrinate	Metazachlor
Carbofuran	Diflufenican	Fludioxinil	Methacrifos
Chlorantraniliprole	Dimethenamid	Flufenoxuron	Methamidophos
Chlorbromuron	Dimethoate	Flumetralin	Methidathion
Chlorfenapyr	Dimethomorph	Fluometuron	Methomyl
Chlorfenvinphos	Dimoxystrobin	Fluotrimazole	Methoxychlor
Chlormephos	Diniconazole	Fluquinconazole	Methoxyfenozide
Chloroneb	Dioxacarb	Flusilazole	Metobromuron
Chloropropylate			

Metolachlor	Pendimethalin	Prometryn	Tefluthrin
Methoprotryne	Pentachloroanisole	Propachlor	Terbacil
Metoxuron	Permethrin	Propamocarb	Terbufos
Metribuzin		Propanil	Terbumeton
Mevinphos	1,1-(2,2-	Propargite	Terbutylazine
Myclobutanil	dichloroethylidene)	Propetamphos	Terbutryn
Molinate	bis(4-	Propiconazole	Tetraconazole
Monocrotophos	methoxybenzene)	Propyzamide	Tetradifon
Monolinuron		Propoxur	Tetramethrin
Monuron	(methoxychlor	Prosulfocarb	Tetrasul
Napropamide	metabolite)	Prothiofos	Thiabendazole
Neburon	Phenmedipham	Pyridafenthion	Thiacloprid
Nitenpyram	Picoxystrobin	Pyrimethanil	Thiamethoxam
Nitrofen	Piperonyl butoxide	Quinalpho	Thiodicarb
Nitrothal-isopropyl	Pyraclostrobin	Quinoxifen	Thiobencarb
Norflurazon	Pyrazphos	Quintozene	Thiometon
Nuarimol	Pyridaben	Rotenone	Tolclofos-methyl
Ofurace	PyrifenoX	Simazine	Triadimefon
Omethoate	Pirimicarb	Simetryn	Triadimenol
op-TDE (DDD)	Pirimicarb-desmethyl	Spinosad A+D	Triazophos
Oxadiazon	Pirimiphos-ethyl	Spirodiclofen	Trichloronate
Oxadixyl	Pirimiphos-methyl	Spiromesifen	Tridemorph
Oxamyl	Pyriproxyfen	Spiroxamine	Trifloxystrobin
Oxamyl-oxime	pp-DDE	Sulfotep	Triflumuron
Oxydemeton-methyl	pp-TDE(DDD)	Sulprofos	Trifluralin
Oxyfluorfen	Prochloraz	Tebuconazole	Vinclozolin
Paclbutrazol	Procymidone	Tebufenozide	Yodofenfos
Parathion	Propham	Tebufenpyrad	Zoxamide
Parathion-methyl	Profenofos	Tebupirimfos	
Pebulate	Profluralin	Tecnazene	
Penconazole	Promecarb	Teflubenzuron	

8. RESULTS EXPRESSION

Each participant laboratory must analyse the sample received according to their routine procedure and fill up the RESULTS form of its client are of the website www.TestQual.com with just one value per analyte/parameter.

The results should be expressed in **µg/Kg**. The number of significant figures and the units are to be chosen by laboratories and will be displayed in the report as received through the website.

The method used for the analysis of each compound informed should be sent when filling up the results form.

The organizer should get the results before the fixed data of the scheme.

If you have any problem logging in to your client area or submitting your results you can contact the coordinator of the PT for guidance or help.

Once the results are sent you can check if they were correctly recorded by accessing the detailed information of this proficiency test, which can be accessed in your client area.

9. STATISTICAL DESIGN

Any participant in this proficiency test can request further information or support about the statistical design that TestQual has developed.

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 by Kernel test, being explained in the final report which is the followed procedure in case there is more than one distribution.

If enough valid results are provided and/or the uncertainty is negligible the **assigned value (X)** will be determined using the robust average of the results considered valid for statistical computing (after eliminating the extreme outliers), according to the standard ISO 13528 into force.

If the number of participants or the number of valid results does not reach a certain number and the uncertainty obtained using robust averages is not negligible, both the assigned value and the uncertainty will be calculated from the spiking/characterization done (design based on ISO 13528 into force).

This is done to evaluate either the assigned value or the results against an independent value from the participants' results, since using a low number of results might cause a bias and to not identify action signals if required. The assigned value obtained from robust statistic is always compared to the assigned value obtained from characterization of the spiking done and depending on the situation TestQual might decide to use either of the assigned value, justifying that decision on the evaluation report.

If the consensus assigned value is not used, then the characterization of the spiking and the uncertainty will be calculated based in a suitable model and according to ISO 13528 into force.

The **standard uncertainty (u_x)** when the assigned value is obtained from the consensus it is calculated using robust statistics from the following formula:

$$u_x = s^*/\sqrt{p}$$

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

When the assigned value is obtained from the spiking done, the uncertainty is calculated accounting all sources of uncertainty according to our internal procedure and according to ISO 13528 into force.

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

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

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 **25 %**.

This value is fixed previously by the organizer based in the experience of TestQual organizing this and similar proficiency tests.

Proficiency assessment (z-score): This score 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 $	≤ 2	Satisfactory
2 <	$ z $	≤ 3	Questionable
	$ z $	> 3	Unsatisfactory

In case the inequation $u_x \leq 0,3 \hat{\sigma}$ is not fulfilled, the participants of the scheme will be informed in the report that the uncertainty is not negligible. For the parameters/analytes in which this situation occurs, the following calculation will be made:

$$z'\text{-score} = (x_i - X) / \sqrt{\hat{\sigma}^2 + U_x^2}$$

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

The criteria for defining the z'-score values are:

	$ z' $	≤ 2	Satisfactory
2 <	$ z' $	≤ 3	Questionable
	$ z' $	> 3	Unsatisfactory

The z'-score is a underestimation of the z-score, for this reason, for those analytes in which the uncertainty of the assigned value cannot be neglected and a z'-score is issued, it will be accompanied by the percentual difference against z-score, this way participants should be able to complete evaluate their performance.

The evaluation could be informative if the difference between scores surpasses the limit contemplated in our procedure. If any analyte or evaluation is informative it will be indicated in the report through marking and a legend.

False negatives: Any analyte not reported in the results that is in the sample above the limit of quantification previously established for this proficiency test by the organization and above the LOQ of the participant laboratory (**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 is not present in the test material, and is reported by the participant at concentrations higher than the limit of quantification of the P.T. (**10 µg/Kg**).

Once the samples are prepared between four and ten of them will be chosen at random and sent to be analysed by TestQual's collaborator laboratory in duplicate in repeatability conditions. The number of samples analysed will be chosen based on: TestQual's experience homogenizing this or similar matrixes, the number of samples analysed is representative to the number of samples prepared in the lot, as well as other factors that if taken into account would be mentioned in the final report. Once received the results, a statistical evaluation will be performed, with a modified calculation from the Harmonic Protocol published by IUPAC and based in ISO 13528 into force.

The acceptance criterion to ensure that the randomly chosen samples are homogeneous is 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, was 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 was 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.

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

In second place to check the criterion for sufficient homogeneity 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 obtained in function of the values from the significance tables: Chi square distribution and F distribution, both with 95% confidence. For example: equal to 1.88 and 1.01 respectively for 10 samples, while it would be 2.1 and 1.43 respectively for 7 samples. The less samples analysed the higher c will be, making harder the criteria to consider the lot homogeneous.

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:

$$\hat{\sigma} = 0.25 \cdot \bar{X}$$

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

If $S_{sam}^2 < c$ is true, then the lot prepared will be considered sufficiently homogeneous and only then it would be distributed. If the results from the homogeneity test does not meet the criteria TestQual would communicate any change required in the proficiency test (new lot will be prepared, new calendar, etc.).

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.

With these values, a study is performed according to the up to date SANTE guide (SANTE/12682/2019 Guidance document on analytical quality control), referred to analysis under repeatability conditions.

The acceptance criteria to ensure the samples have been stable during the whole proficiency test are the following:

$$\begin{aligned} |(X_{t1} - X_{t2}) / X_{t1}| \cdot 100 &\leq 10\% \\ |(X_{t1} - X_{t3}) / X_{t1}| \cdot 100 &\leq 10\% \end{aligned}$$

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 be received by the laboratories via e-mail in PDF format or an email notifying that the report is now available to be downloaded from the private area of each participant in www.TestQual.com.

If desired, the laboratory may request the report in paper, and it will be sent to its laboratory by mail.

In the event that a participant wishes to appeal against the assessment program performance, a written appellation must be sent by e-mail to jpnavarro@testqual.com explaining the reasons for it.

11. CONTACT

TestQual puts at your disposal any of the following means to contact our team:

Website:	Contact
Email:	jpnavarro@testqual.com
Office phone:	+34 868 94 94 86
Mobile phone:	+34 676 367 555

12. 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

EU Pesticides database (v.2.2) Search Pesticides residues

SANTE/12682/2019, 1st January 2020, Guidance document on analytical quality control and method validation procedures for pesticides residues analysis in food and feed.

END OF DOCUMENT

