TESTQUAL
PROFICIENCY TESTING SCHEMES

TestQual, S.L.

(Proficiency Testing Schemes)

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FINAL REPORT TestQual 204

Multiresidue Pesticides, fosetyl, phosphonic acid, AMPA and glyphosate in Avocado

LABORATORY CODE:

To check your laboratory code login in your client area on www.**TestQual**.com, request a participation certificate or your individual report.

ISSUE DATE OF THE REPORT: 2025/01/20

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Ana María Moreno López

Technical department of **TestQual, S.L.**

0. GLOSARY AND ABREVIATIONS

Text	Abbreviation
TestQual	TQ
Proficiency test	PT / P.T.
Limit Of Quantification	LOQ
NA	Not Analysed
Decimal separator	,
$_t$RSD	Target Relative Standard Deviation
Consensus	Consens.
Formulation/Characterization	Formu.
Quaternary Ammonium Compounds	QAC
DidecylDimethylAmmonium Chlorides	DDAC
AlkylBenzyldimethyAmmonium Chlorides	BAC
PolyChlorinated Biphenyl /s	PCB / PCBs
Aminomethylphosphonic acid	AMPA
Fosetyl-Al (Sum of fosetyl and phosphonic acid and its salts expressed as fosetyl)	Fosetyl-Al (sum)

SUMMARY

The samples from this proficiency test were sent in December of 2024 to ***11*** participant laboratories and ***all of them*** sent their results.

Summary **TestQual** 204 - Avocado results:

ANALYTE	ACCREDITED ?	ASSIGNED VALUE ORIGIN	EVALUATION OF THE ASSIGNED VALUE	NUMBER OF DATA*	ASSIGNED VALUE (µg/kg)	UNCERTAINTY (µg/kg)	%$_t$RSD	TARGET STANDARD DEVIATION (µg/kg)	ROBUST STANDARD DEVIATION (µg/kg)
FOSETYL	NO	Consens.	-	8	48,96	3,86	25	12,24	10,91
PHOSPHONIC ACID	NO	Consens. **	-	6	129,61	15,75	25	32,40	38,58
FOSETYL-AL (SUM)	NO	Formu.	OK	7	178,78	5,05	25	44,69	69,78
GLYPHOSATE	NO	Formu.	OK	6	85,86	2,75	25	21,47	19,80
AMPA	NO	Formu.	OK	5	71,27	4,46	25	17,82	20,66
CIPROCONAZOLE	NO	Consens.	-	10	28,00	2,18	25	7,00	6,90
CYPRODINIL	NO	Consens.	OK	9	44,62	3,68	25	11,16	11,04
PENDIMETHALIN	NO	Consens.	OK	10	69,90	5,43	25	17,47	17,18
IMIDACLOPRID	NO	Consens.	OK	8	62,20	5,86	25	15,55	16,58

ANALYTE	ACCREDITED ?	ASSIGNED VALUE ORIGIN	EVALUATION OF THE ASSIGNED VALUE	NUMBER OF DATA*	ASSIGNED VALUE (µg/kg)	UNCERTAINTY (µg/kg)	%tRSD	TARGET STANDARD DEVIATION (µg/kg)	ROBUST STANDARD DEVIATION (µg/kg)
PYRIPROXYFEN	NO	Consens.	OK	9	63,84	5,49	25	15,96	16,48

**Results considered extreme outliers have not been considered*

*** The consensus value is given for information purposes, as the minimum number of results required by **TestQual** (eight) was not reached.*

ANALYTE	Z / Z' SCORE	NUMBER OF SCORES*	% SATISFACTORY	% QUESTIONABLE	% UNSATISFACTORY
FOSETYL	Z'	9	89	0	11
PHOSPHONIC ACID	Z'	8	88	13	0
FOSETYL-AL (SUM)	z	9	78	0	22
GLYPHOSATE	z	7	86	0	14
AMPA	z	7	57	14	29
CIPROCONAZOLE	Z'	10	100	0	0
CYPRODINIL	Z'	9	100	0	0
PENDIMETHALIN	Z'	10	100	0	0
IMIDACLOPRID	Z'	9	89	11	0
PYRIPROXYFEN	Z'	9	100	0	0

**Every result has received an evaluation (z-score or z'-score), including the results considered as extreme outliers.*

There are PT items available from this PT as Quality Control Material and can be acquired from **TestQual**'s website.

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1. OBJECTIVE AND CALENDAR

The aim of the **TestQual 204 Avocado** Proficiency Test is to gather information about the quality and accuracy of the results sent by the participating laboratories.

This proficiency test is based on the analysis of **multiresidue pesticides, fosetyl, phosphonic acid, glyphosate and AMPA** in **avocado**. After the evaluation of the applications (depending on the LOQ of the laboratory and the checking the participant will receive the sample correctly), **11** laboratories were accepted, and the test material was sent in **December of 2024**. The assigned concentration value (µg/kg) for the analyte present in the sample was calculated by consensus among participating laboratories.

The laboratory results were considered satisfactory if the z-score parameter was $|score| \leq 2$, questionable if $2 \leq |score| \leq 3$ and unsatisfactory if $|score| > 3$.

The most important dates of the proficiency test have been:

Date	Activity	Carried out by
2024/11/18	Deadline to send application* (acceptance depends on availability)	Participants
2024/12/02-04	Sample delivery	**TestQual**
2024/12/20	Final date to submit AMPA results	Participants
2024/12/23	Final date to submit rest of results	Participants
2025/01/20	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.

Program coordinators: María Dolores García Valiente/ Ana María Moreno López

Each laboratory was assigned a unique code to participate in the proficiency test. These codes were only known by the laboratory and **TestQual**, and they were confidential during and after the proficiency test.

If any participant wants to appeal against the evaluation of their performance, their allegations must be sent by mail to testqual@testqual.com.

2. TEST MATERIAL. DISTRIBUTION AND CONTROL.

TestQual 204 scheme is a proficiency test based in the analysis of **multiresidue pesticides, fosetyl, phosphonic acid, glyphosate and AMPA** in **avocado** that has been spiked with:

STANDARD
FOSETYL
PHOSPHONIC ACID
GLYPHOSATE
AMPA
CIPROCONAZOLE
CYPRODINIL
PENDIMETHALIN
IMIDACLOPRID
PYRIPROXYFEN

About **9,5** Kg of matrix was bought in a specialized shop in Spain.

The material was cut in very small pieces, and once it was fully frozen, it was grounded into a fine powder, puree or juice, which was spiked with a solution with the analytes of the proficiency test and poured into a homogenizer with controlled temperature to ensure complete homogeneity.
Once the sample batch was ready the sample were stored in a temperature-controlled freezer below -20ºC until the dispatch of the samples.

Each sample had approximately **150 g.** Once the sample batch was ready the samples were stored until distribution.

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 were distributed, for the assessment of the homogeneity of the sample batch that was prepared, ten samples from the lot were selected randomly and analysed in duplicate by **TestQual**´s collaborator laboratory under repeatability conditions.

Once ensured the homogeneity of the samples, these were sent to the participants by express courier, under the proper conditions of temperature and conservation.

For stability assessment purpose, three samples are analysed, in duplicate, before, during and at the end (once all laboratories had sent their 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.

Later in this report can be found the conclusions of these tests, all participants of this PT have available upon request the results and calculations done.

3. ANALYSIS

Each participant had to analyse the sample, detect and quantify the presence of **multiresidue pesticides, fosetyl, phosphonic acid, glyphosate and AMPA** in the test material according to their own procedures. Then, fill in with just one result per analyte the "Results" Form of its Private Area of the website *www.**TestQual**.com*, expressing the results in **µg/kg**.

The techniques and analysis method used were chosen by the laboratories, and they are shown later in this report.

4. STATISTICAL RESULTS EVALUATION

The number of significant figures and the units are shown as they were submitted by the laboratories.

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

TestQual consider as an **extreme outlier** any data which differs more than **50 %** to the median of all results reported by the laboratories. These extreme values are not considered for the calculation of the assigned value.

If there are enough results the **assigned value (X)** was determined by consensus using the robust average of the results considered valid for statistical computing (after removing extreme outliers), according to the standard ISO/IEC 13528 into force.

The **standard uncertainty (u_x)**(consensus) was 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 not considered as extreme outliers.

If the number of participants or the number of valid results does not reach the minimum accepted by **TestQual** (eight) the evaluation of the analyte will be issued not accredited. However, due to special and temporary circumstances, the report is issued without accreditation.

If the uncertainty of the assigned value (consensus) cannot be neglected, and depending how significant it is, the evaluation will be issued using z'-score or informative.

When only a few results would be available, the evaluation through consensus would be highly dependent on the results themselves (even with robust statistic), possible causing the evaluation to suffer a bias and do not issue action signals if required.

If the uncertainty of the assigned value (consensus) cannot be neglected and eight or fewer results are available, the evaluation will be done against the known value, obtaining the assigned value and its uncertainty through formulation/characterization (calculation design based in ISO 13528 into force). Being only accepted if it does not differ greatly from the available consensus value. The formulation value would be rejected if the difference between the characterization and the consensus value is greater than what our internal procedure determines.

If a significative difference is detected between consensus and characterization assigned values, **TestQual** would issue the evaluation either with consensus or characterization with informative value, detailing in the report the reasons.

Likewise, the assigned value obtained from consensus is always compared against the formulation done,

ensuring there is not a significative difference between them, to offer the evaluation with the utmost confidence. Would a great difference be obtained between these two, it would be explained in the evaluation report and could include conclusions or thoughts about how this could have happened.

The following condition must be fulfilled to discard the contribution of the uncertainty and neglect it:

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

In case this condition is not fulfilled, the participants of the scheme will be informed in the report that the uncertainty of an assigned value is not negligible. For the parameters/analytes in which this situation occurs, the evaluation will be issued as z'-score according to the following calculation:

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

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 Ux is the uncertainty of the assigned value.
If applies and the evaluation meets the requirements of our technical annex, z'-score will be issued accredited.

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

	$\lvert z' \rvert$	≤2	Satisfactory
2 <	$\lvert z' \rvert$	≤3	Questionable
	$\lvert z' \rvert$	>3	Unsatisfactory

The z'-score is an 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.

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

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

Being $b_i = \%_{RSDA}/100$, and $\%_{RSDA}$ is the assigned **relative standard deviation**.
In this case, the assigned relative standard deviation is **25 %**.
This value was previously set by the organizer and informed in the protocol of the proficiency test, based on the extensive experience of **TestQual** organizing this and 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 each of the laboratories, X is the assigned value, and $\hat{\sigma}$ is the target standard deviation for each analyte.

The criterion for defining the z-score values is:

	$\lvert Z \rvert$	≤ 2	Satisfactory
$2 <$	$\lvert Z \rvert$	≤ 3	Questionable
	$\lvert Z \rvert$	> 3	Unsatisfactory

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, and are reported by the participant at concentrations higher than the limit of quantification of the proficiency test **(10 µg/kg).**

Testing for sufficient homogeneity:

Ten samples were chosen at random and sent to be analysed by **TestQual**'s subcontracted laboratory in duplicate in repeatability conditions.

Once received the results, a statistical evaluation was performed, with the homogeneity evaluation of 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:

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

Being S_{sam}^2 the estimated sampling standard deviation, obtained from the variance of the results sums and the experimental estimate of analytical standard deviation (S_{an}), which in turn is obtained from the differences between replicates of the same sample. Lastly, *c* is the limit value, which is obtained as ISO 13528 calculations and it considers two constants, obtained from two significance groups, which are multiplied with a term related to the target standard deviation and the S_{an}.
If $S_{sam}^2 < c$ is true, then the lot prepared will be considered sufficiently homogeneous and only then it would be distributed. With this test what we achieve is to check if the intra-sample deviation is lower than the inter-sample deviation.

If the results from the homogeneity test did 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 were analysed, in duplicate, before, during and at the end (once all laboratories have submitted their results) of the proficiency test. The acceptance criteria to ensure the samples have been stable during the proficiency test are the following:

$$\left|\frac{X_{t1} - X_{t2}}{X_{t1}}\right| \cdot 100 \leq 10\%$$

$$\left|\frac{X_{t1} - X_{t3}}{X_{t1}}\right| \cdot 100 \leq 10\%$$

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

*The conclusions of these tests are shown later in this report.

Checking unimodality of the results:

The Kernel density representation can be used to evaluate if the results employed to calculate the assigned value are from different distributions or not, this representation is a non-parametric estimation that represents the population density function versus the data results not considered as outliers. The smoothing parameter or bin width *h* was selected as $h=0.75 \cdot \hat{\sigma}$ as referred in ISO 13528 into force. Through the evaluation of the symmetry of this distribution it is possible to evaluate the unimodality of the data set. These representations and the conclusions drawn can be found in later in this report.

5. RESULTS

5.1. RESULTS, LIMITS OF QUANTIFICATION AND Z-SCORE

Legend:

FN:	Analyte no informed (not detected) by the participant (false negative).
<LOQ:	Analyte in a concentration lower than the participant's limit of quantification. Possible false negative if the analyte was present.
*[A]:	Result considered as statistically aberrant and not considered to calculate the assigned value.
Bold:	Results with z-score≥|2|.
% DIFF.:	Difference between z-score and z'-score, degree in which the latter is an underestimation of the performance.
Xf:	Assigned value (formulation).
Xc:	Assigned value (consensus).
Ux:	Uncertainty of the assigned value with 95% confidence.
X_i:	Participant's reported value.
LOQ:	Participant's Limit of quantification.
NA:	Analyte not analysed by the participant.
$\hat{\sigma}$:	Target standard deviation

	FOSETYL			PHOSPHONIC ACID			FOSETYL-AL (SUM)			GLYPHOSATE		
	(Xc= 48,96 µg/kg)			(Xc= 129,61 µg/kg)			(Xf= 178,78 µg/kg)			(Xf= 85,86 µg/kg)		
	(Ux= 3,86 µg/kg)			(Ux= 15,75 µg/kg)			(Ux= 5,05 µg/kg)			(Ux= 2,75 µg/kg)		
	($\hat{\sigma}$= 12,24 µg/kg)			($\hat{\sigma}$= 32,40 µg/kg)			($\hat{\sigma}$= 44,69 µg/kg)			($\hat{\sigma}$= 21,47 µg/kg)		
	% DIFF. 4,63		z-score	% DIFF. 10,06		z-score	% DIFF. -		z-score	% DIFF. -		z-score
LABORATORY CODE	Xi µg/kg	LOQ µg/kg	Z'-score	Xi µg/kg	LOQ µg/kg	Z'-score	Xi µg/kg	LOQ µg/kg	z-score	Xi µg/kg	LOQ µg/kg	z-score
TQ24-0204-001	NA			NA			NA			NA		
TQ24-0204-002	64	10	1,2	143	10	0,4	253	10	1,7	NA		
TQ24-0204-003	56,3	10	0,6	104,3	10	-0,7	190,2	10	0,3	96,4	10	0,5
TQ24-0204-004	55	10	0,5	145	10	0,4	249,3	10	1,6	**175*A**	**10**	**4,2**
TQ24-0204-005	36	20	-1,0	**FN**	**1000**	**<LC**	**33*A**	**20**	**-3,3**	**FN**	**100**	**<LC**
TQ24-0204-006	NA			NA			NA			NA		
TQ24-0204-007	**337*A**	**10**	**22,4**	170	20	1,1	**565*A**	**10**	**8,6**	105	10	0,9
TQ24-0204-008	40	10	-0,7	**54*A**	**10**	**-2,1**	112	10	-1,5	61	10	-1,2
TQ24-0204-009	45	10	-0,3	60*A	20	-1,9	125	20	-1,2	65	10	-1,0
TQ24-0204-010	53,35	10	0,3	140,36	10	0,3	242	10	1,4	74,73	10	-0,5
TQ24-0204-011	42	10	-0,5	75	10	-1,5	142,5	10	-0,8	85	10	0,0

	AMPA			CIPROCONAZOLE			CYPRODINIL		
	(Xf= 71,27 µg/kg)			(Xc = 28,00 µg/kg)			(Xc = 44,62 µg/kg)		
	(Ux= 4,46 µg/kg)			(Ux= 2,18 µg/kg)			(Ux= 3,68 µg/kg)		
	($\hat{\sigma}$= 17,82 µg/kg)			($\hat{\sigma}$= 7,00 µg/kg)			($\hat{\sigma}$= 11,16 µg/kg)		
	% DIFF. -		z-score	% DIFF. 4,54		z-score	% DIFF. 5,03		z-score
LABORATORY CODE	Xi µg/kg	LOQ µg/kg	z-score	Xi µg/kg	LOQ µg/kg	Z'-score	Xi µg/kg	LOQ µg/kg	Z'-score
TQ24-0204-001	NA			30	10	0,3	52	10	0,6
TQ24-0204-002	NA			22	10	-0,8	37	10	-0,6
TQ24-0204-003	**230*A**	**10**	**8,9**	36,6	10	1,2	56,1	10	1,0
TQ24-0204-004	**35,3*A**	**10**	**-2,0**	35	10	1,0	45	10	0,0
TQ24-0204-005	65	100	-0,4	27	10	-0,1	57	10	1,1
TQ24-0204-006	NA			34	30	0,8	NA		
TQ24-0204-007	NA			30	5	0,3	24	5	-1,8
TQ24-0204-008	72,5140	10	0,1	22	10	-0,8	46	10	0,1
TQ24-0204-009	**FN**	**10**	**-3,7**	19	10	-1,2	45	10	0,0
TQ24-0204-010	77,74	10	0,4	24,433	10	-0,5	35,458	10	-0,8
TQ24-0204-011	45	0,10	-1,5	NA			NA		

	PENDIMETHALIN			IMIDACLOPRID			PYRIPROXYFEN		
	(Xc = 69,90 µg/kg)			(Xc = 62,20 µg/kg)			(Xc = 63,84 µg/kg)		
	(Ux= 5,43 µg/kg)			(Ux= 5,86 µg/kg)			(Ux= 5,49 µg/kg)		
	($\hat{\sigma}$= 17,47 µg/kg)			($\hat{\sigma}$= 15,55 µg/kg)			($\hat{\sigma}$= 15,96 µg/kg)		
	% DIFF. 4,51		z-score	% DIFF. 6,43		z-score	% DIFF. 5,44		z-score
LABORATORY CODE	Xi µg/kg	LOQ µg/kg	Z'-score	Xi µg/kg	LOQ µg/kg	Z'-score	Xi µg/kg	LOQ µg/kg	Z'-score
TQ24-0204-001	94	10	1,3	63	10	0,0	82	10	1,1
TQ24-0204-002	53	10	-0,9	78	10	1,0	43	10	-1,2
TQ24-0204-003	68,8	10	-0,1	62,3	10	0,0	66,7	10	0,2
TQ24-0204-004	50	10	-1,1	50	10	-0,7	60	10	-0,2
TQ24-0204-005	87	10	0,9	44	10	-1,1	NA		
TQ24-0204-006	74	30	0,2	NA			72	30	0,5
TQ24-0204-007	66	5	-0,2	**25*[A]**	**5**	**-2,2**	37	10	-1,6
TQ24-0204-008	80	10	0,6	80	10	1,1	65	10	0,1
TQ24-0204-009	75	10	0,3	75	10	0,8	68	10	0,2
TQ24-0204-010	51,191	10	-1,0	45,32	10	-1,0	78,53	10	0,9
TQ24-0204-011	NA			NA			NA		

5.3. PARTICIPANTS AND TESTQUAL COMMENTS

No comments from participants.

TestQual indicates:

- TQ24-0204-005 has declared an LOQ of 100 µg/kg for AMPA but still reported a value of 65 µg/kg with which it wants to participate.
- The value assigned for phosphonic acid, although obtained by consensus, is given for information only, as it does not reach the minimum number of results required by **TestQual**.
- TQ24-0204-003 reports a CS% of 126% for AMPA.

5.4. FALSE POSITIVES AND FALSE NEGATIVES

5.4.1. FALSE POSITIVES:

No false positives were reported for this proficiency test.

5.4.2. FALSE NEGATIVES:

Laboratory	Analyte (µg/kg)	LOQ (µg/kg)	Assigned value (µg/kg)
TQ24-0204-005	PHOSPHONIC ACID	1000	129,61
TQ24-0204-005	GLYPHOSATE	100	85,86
TQ24-0204-009	AMPA	10	71,27

5.5. ASSIGNED VALUE AND TARGET STANDARD DEVIATION

ANALYTE	ACCREDITED ?	ASSIGNED VALUE ORIGIN	EVALUATION OF THE ASSIGNED VALUE	NUMBER OF DATA*	ASSIGNED VALUE (µg/kg)	UNCERTAINTY (µg/kg)	%tRSD	TARGET STANDARD DEVIATION (µg/kg)	ROBUST STANDARD DEVIATION (µg/kg)
FOSETYL	NO	Consens.	-	8	48,96	3,86	25	12,24	10,91
PHOSPHONIC ACID	NO	Consens. **	-	6	129,61	15,75	25	32,40	38,58
FOSETYL-AL (SUM)	NO	Formu.	OK	7	178,78	5,05	25	44,69	69,78
GLYPHOSATE	NO	Formu.	OK	6	85,86	2,75	25	21,47	19,80
AMPA	NO	Formu.	OK	5	71,27	4,46	25	17,82	20,66
CIPROCONAZOLE	NO	Consens.	-	10	28,00	2,18	25	7,00	6,90
CYPRODINIL	NO	Consens.	OK	9	44,62	3,68	25	11,16	11,04
PENDIMETHALIN	NO	Consens.	OK	10	69,90	5,43	25	17,47	17,18
IMIDACLOPRID	NO	Consens.	OK	8	62,20	5,86	25	15,55	16,58
PYRIPROXYFEN	NO	Consens.	OK	9	63,84	5,49	25	15,96	16,48

**Results considered extreme outliers have not been considered*

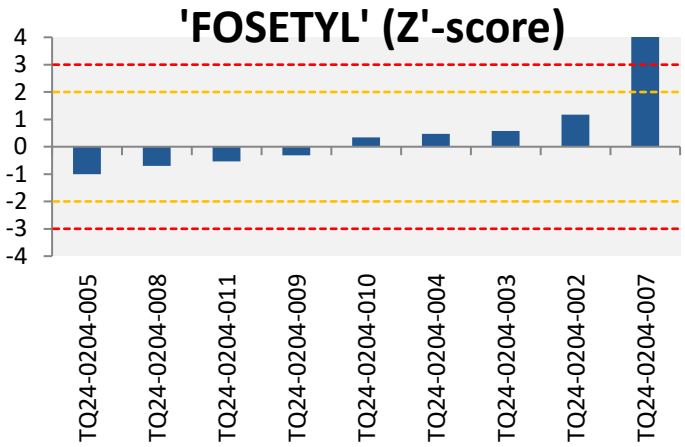
*** The consensus value is given for information purposes, as the minimum number of results required by **TestQual** (eight) was not reached.*

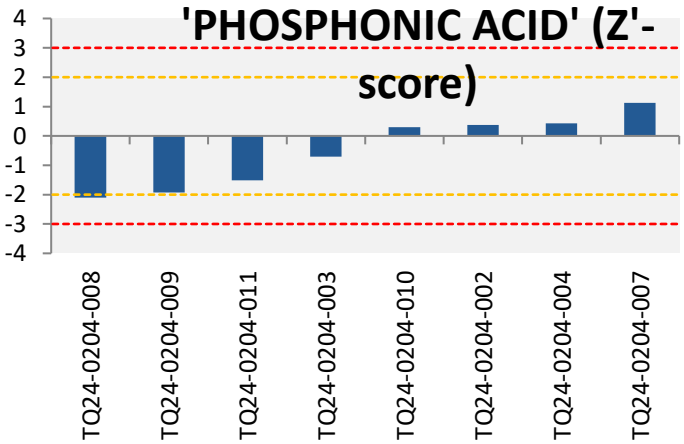
5.6. SATISFACTORY, QUESTIONABLE AND UNSATISFACTORY Z-SCORES

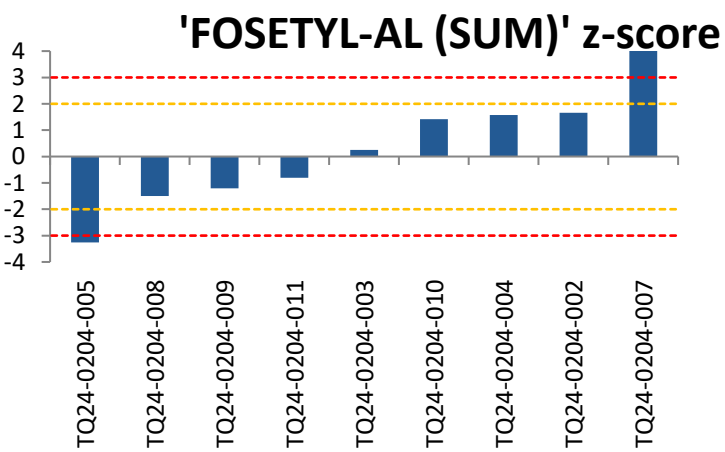
ANALYTE	Z / Z' SCORE	NUMBER OF SCORES*	% SATISFACTORY	% QUESTIONABLE	% UNSATISFACTORY
FOSETYL	Z'	9	89	0	11
PHOSPHONIC ACID	Z'	8	88	13	0
FOSETYL-AL (SUM)	z	9	78	0	22
GLYPHOSATE	z	7	86	0	14
AMPA	z	7	57	14	29
CIPROCONAZOLE	Z'	10	100	0	0
CYPRODINIL	Z'	9	100	0	0
PENDIMETHALIN	Z'	10	100	0	0
IMIDACLOPRID	Z'	9	89	11	0
PYRIPROXYFEN	Z'	9	100	0	0

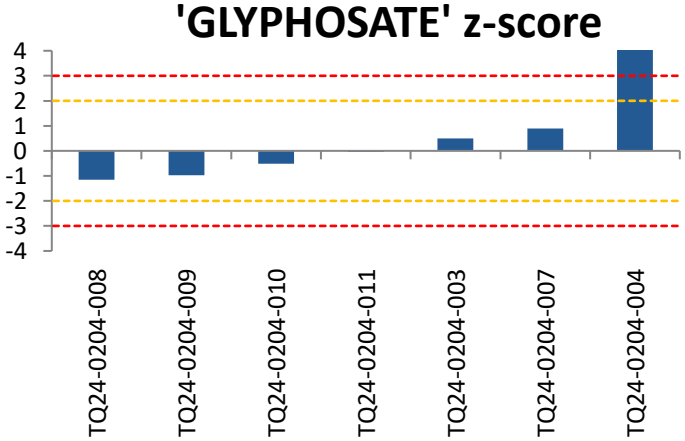
**Every result has received an evaluation (z-score or z'-score), including the results considered as extreme outliers.*

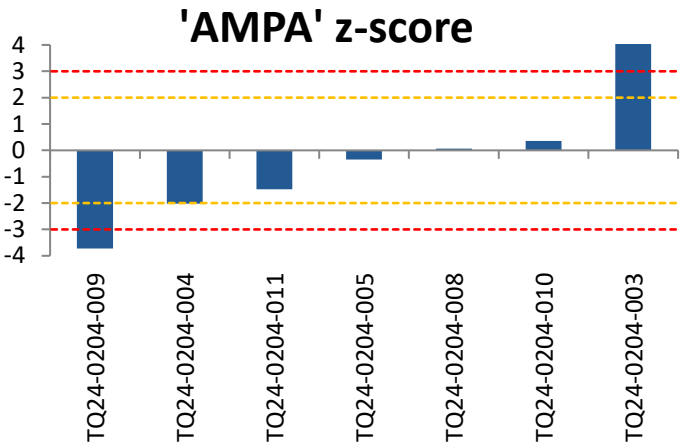
6. GRAPHICAL REPRESENTATION OF ASSIGNED Z-SCORES VALUES

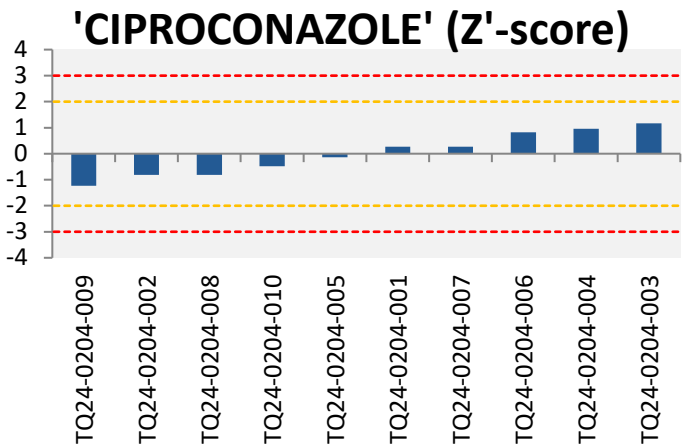


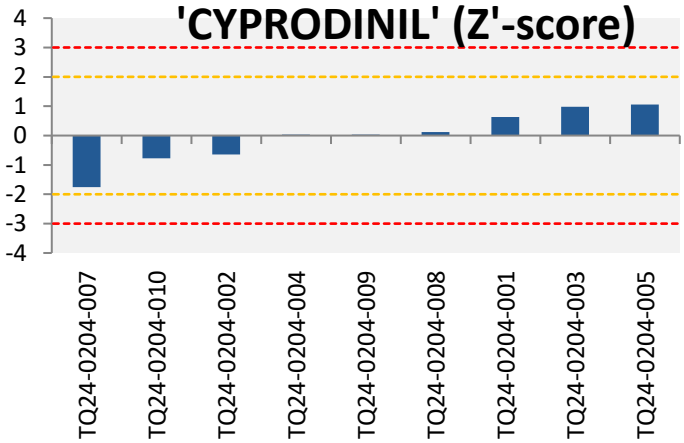


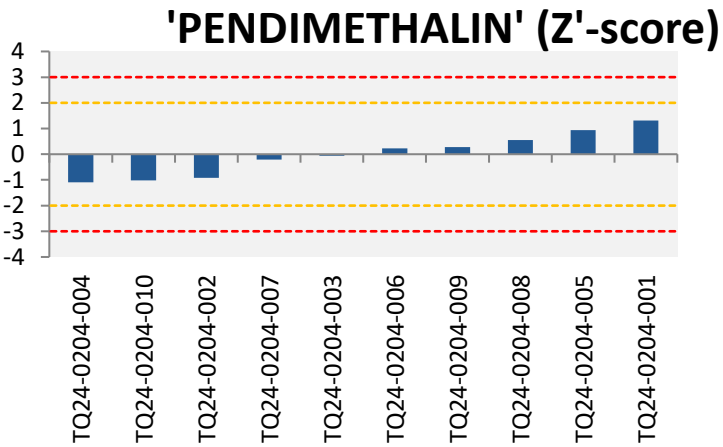


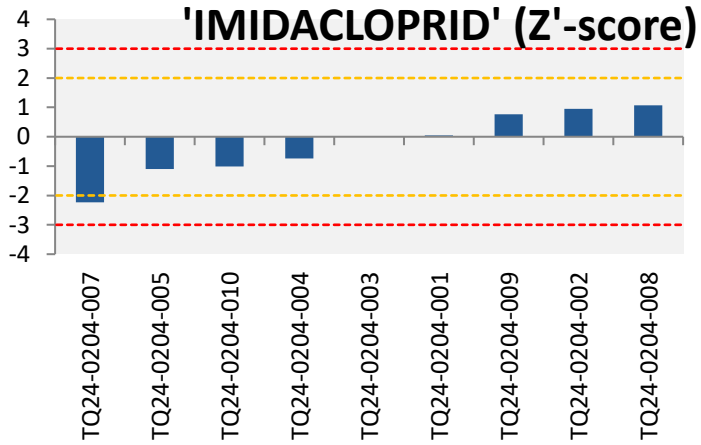


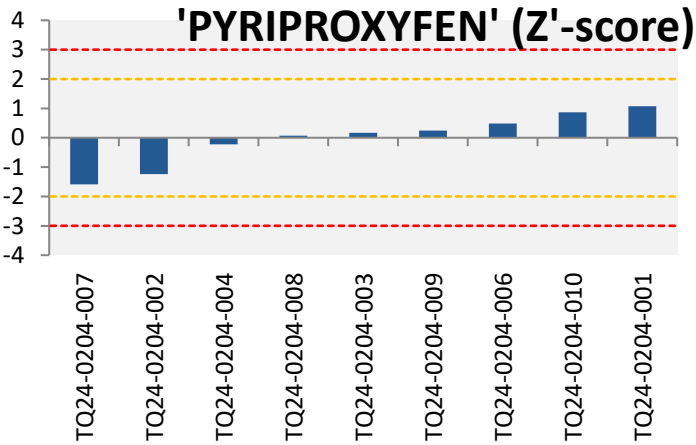

'AMPA' z-score
4
3
2
1
0
-1
-2
-3
-4
TQ24-0204-009
TQ24-0204-004
TQ24-0204-011
TQ24-0204-005
TQ24-0204-008
TQ24-0204-010
TQ24-0204-003


'CIPROCONAZOLE' (Z'-score)
4
3
2
1
0
-1
-2
-3
-4
TQ24-0204-009
TQ24-0204-002
TQ24-0204-008
TQ24-0204-010
TQ24-0204-005
TQ24-0204-001
TQ24-0204-007
TQ24-0204-006
TQ24-0204-004
TQ24-0204-003


'CYPRODINIL' (Z'-score)
4
3
2
1
0
-1
-2
-3
-4
TQ24-0204-007
TQ24-0204-010
TQ24-0204-002
TQ24-0204-004
TQ24-0204-009
TQ24-0204-008
TQ24-0204-001
TQ24-0204-003
TQ24-0204-005


'PENDIMETHALIN' (Z'-score)
4
3
2
1
0
-1
-2
-3
-4
TQ24-0204-004
TQ24-0204-010
TQ24-0204-002
TQ24-0204-007
TQ24-0204-003
TQ24-0204-006
TQ24-0204-009
TQ24-0204-008
TQ24-0204-005
TQ24-0204-001


'IMIDACLOPRID' (Z'-score)
4
3
2
1
0
-1
-2
-3
-4
TQ24-0204-007
TQ24-0204-005
TQ24-0204-010
TQ24-0204-004
TQ24-0204-003
TQ24-0204-001
TQ24-0204-009
TQ24-0204-002
TQ24-0204-008


'PYRIPROXYFEN' (Z'-score)
4
3
2
1
0
-1
-2
-3
-4
TQ24-0204-007
TQ24-0204-002
TQ24-0204-004
TQ24-0204-008
TQ24-0204-003
TQ24-0204-009
TQ24-0204-006
TQ24-0204-010
TQ24-0204-001

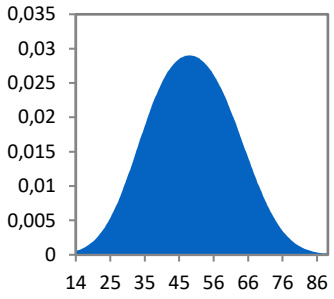
7. RESULTS DISTRIBUTION (KERNEL DENSITY):

For this proficiency test, all analytes except AMPA and fosetyl-Al have shown adequate symmetry to be considered unimodal.

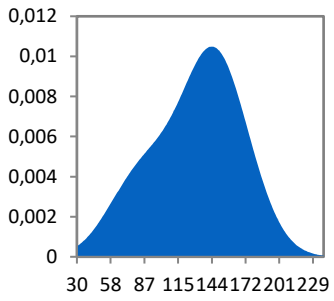
We have at the participant's disposal this same graphics with more detail and size.

X axis: Results (µg/kg); Y axis: Density

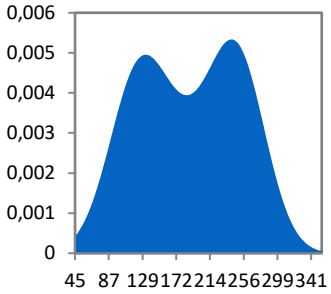
'FOSETYL'
Fixed h=9,2



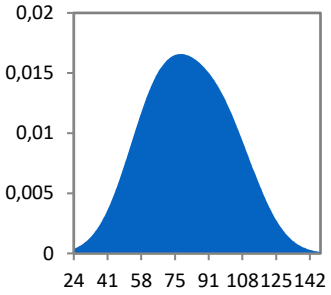
'PHOSPHONIC ACID'
Fixed h=24,3



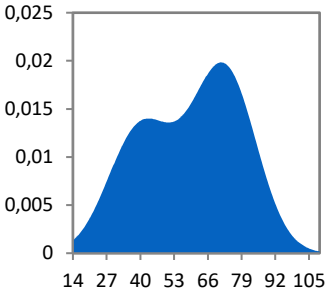
'FOSETYL-AL (SUM)'
Fixed h=35,2



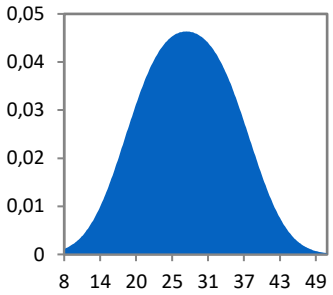
'GLYPHOSATE'
Fixed h=15,2



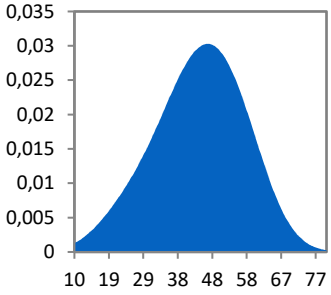
'AMPA'
Fixed h=11,1



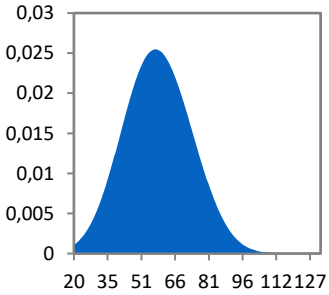
'CIPROCONAZOLE'
Fixed h=5,3



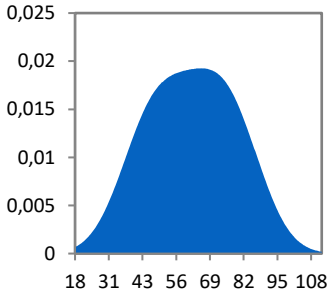
'CYPRODINIL'
Fixed h=8,4

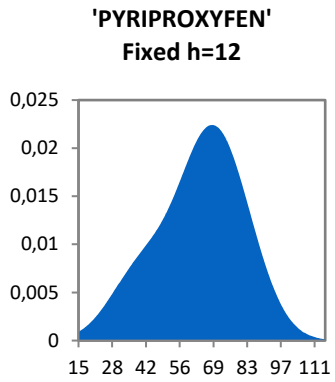


'PENDIMETHALIN'
Fixed h=13,1



'IMIDACLOPRID'
Fixed h=11,7





After checking the data provided by the laboratories regarding their methods used for the analysis of these parameters and after verifying that our homogeneity analyses were successfully passed:

Fosetyl-Al → A bimodality appears in the results which may be due to differences between laboratories when expressing results for this analyte or the use of different methods by the participating laboratories. This bimodality is not due to a lack of homogeneity as our homogeneity checks have been successfully passed.

AMPA → A bimodality appears in the results which may be due to differences between laboratories when expressing results for this analyte or the use of different methods by the participating laboratories. This bimodality is not due to a lack of homogeneity as our homogeneity checks have been successfully passed.

8. HOMOGENEITY AND STABILITY OF THE TEST MATERIAL

To ensure the evaluation issued is useful, the sample batch goes through two different kind of quality controls:

1. The homogeneity checks to ensure the difference between-sample is not greater than the in-between variation of a single sample.
2. The stability check, used to ensure any change in the concentration does not affect, or affects below a certain point, the score obtained by each participant. The analysis of the stability is done before and after sending the samples to the participants plus a last time after all results have been received.

The data employed to check the homogeneity are 20 results, all obtained in repeatability conditions from a set of randomized samples from the lot prepared, while the stability check is obtained from a total of 6 results, 2 from the initial concentration, 2 after sending the samples to the participants and the last 2 results after all results have been received. All the results used to check the criteria were not considered as absolute concentrations, only as relative data for the criteria calculation.

These results were analysed according to calculations on section 4 of this report, and these are the conclusion reached:

	Analyte	FOSETYL	PHOSPHONIC ACID	FOSETYL-AL (SUM)	GLYPHOSATE	AMPA
HOMOGENEITY	$c < 0.3*\sigma$	Satisfies criteria	Satisfies criteria	Satisfies criteria	Satisfies criteria	Satisfies criteria
STABILITY	$\|Difference\| \leq 10\%$ t_2 vs t_1	Does not satisfy criteria	Does not satisfy criteria	Does not satisfy criteria	Does not satisfy criteria	Satisfies criteria
	$\|Difference\| \leq 10\%$ t_3 vs t_1	Does not satisfy criteria	Does not satisfy criteria	Does not satisfy criteria	Does not satisfy criteria	Satisfies criteria

	Analyte	CIPROCONAZOLE	CYPRODINIL	PENDIMETHALIN	IMIDACLOPRID	PYRIPROXYFEN
HOMOGENEITY	$c < 0.3*\sigma$	Satisfies criteria	Satisfies criteria	Satisfies criteria	Satisfies criteria	Satisfies criteria
STABILITY	$\|Difference\| \leq 10\%$ t_2 vs t_1	Satisfies criteria	Satisfies criteria	Satisfies criteria	Does not satisfy criteria	Satisfies criteria
	$\|Difference\| \leq 10\%$ t_3 vs t_1	Does not satisfy criteria	Satisfies criteria	Satisfies criteria	Does not satisfy criteria	Satisfies criteria

Fosetyl → Upon looking at the experimental robust standard deviation (22,29%) against the target standard deviation (25%) we can issue the evaluation with confidence, since the participants did not suffer from the detected instability by the collaborator laboratory.

Phosphonic acid → Despite not meeting the stability criteria, based on the results obtained and **TestQual**'s experience in this type of testing, we do not question the confidence of the assessment issued. However, the stability non-conformance is being investigated with the collaborating laboratory. Although we do not reach the minimum number of participants required by **TestQual** to obtain a consensus value, we offer this for information purposes in order to give a more confident assessment based on the results obtained.

Fosetyl-Al → Despite not meeting the stability criteria, based on the results obtained and **TestQual**'s experience in this type of testing, we do not question the confidence of the evaluation of the results. However, this non-stability is being investigated with the collaborating laboratory.

Glyphosate → Upon looking at the experimental robust standard deviation (24,39%) against the target standard deviation (25%) we can issue the evaluation with confidence, since the participants did not suffer from the detected instability by the collaborator laboratory.

Ciproconazole → It does not meet the established 10% between replicates in the case of the last stability but it does meet the 20% of replicates in reproducibility allowed by the SANTE guidelines, so we do not question the confidence of the assessment. Moreover, upon looking at the experimental robust standard deviation (24,66%) against the target standard deviation (25%) we can issue the evaluation with confidence, since the participants did not suffer from the detected instability by the collaborator laboratory.

Imidacloprid → It does not meet the 10% established between replicates but does meet the 20% of replicates in reproducibility allowed by the SANTE guidelines, so we can issue the evaluation with confidence, since the participants did not suffer from the detected instability by the collaborator laboratory.

9. ANALYTICAL METHODS USED BY THE LABORATORIES

FOSETYL						
LABORATORY CODE	ACCREDITED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ24-0204-001	YES.	10	ACN	.	Matrix-matched – Internal standard	HPLC-MS/MS
TQ24-0204-002	-	-	-	-	-	-
TQ24-0204-003	YES. FAL_01_B	2	H2O+MeOH	. In House Method	Solvent - Internal standard	HPLC-MS/MS. FAL_01_B
TQ24-0204-004	NO.	5	Acidified MeOH	. QuPPe	Solvent – Internal standard	HPLC-MS/MS
TQ24-0204-005	YES. SOP No.81	10	Methanol	Extraction with solvents.	Matrix-matched – External standard	HPLC-MS/MS
TQ24-0204-006	-	-	-	-	-	-
TQ24-0204-007	YES.	5	methanol	. QuPPe	Matrix-matched – External standard	HPLC-MS/MS
TQ24-0204-008	YES. PE-6015	10	Methanol/Water	.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-009	YES.	10	methanol	.	Matrix-matched – Internal standard	HPLC-MS/MS
TQ24-0204-010	-	-	-	-	-	-
TQ24-0204-011	-	-	-	-	-	-

PHOSPHONIC ACID						
LABORATORY CODE	ACCREDITED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ24-0204-001	-	-	-	-	-	-
TQ24-0204-002	YES.	10	ACN	.	Matrix-matched – Internal standard	HPLC-MS/MS
TQ24-0204-003	YES. FAL_01_B	2	H2O+MeOH	. In House Method	Solvent - Internal standard	HPLC-MS/MS. FAL_01_B
TQ24-0204-004	NO.	5	Acidified MeOH	. QuPPe	Solvent – Internal standard	HPLC-MS/MS
TQ24-0204-005	-	-	-	-	-	-
TQ24-0204-006	-	-	-	-	-	-
TQ24-0204-007	YES.	5	methanol	. QuPPe	Matrix-matched – External standard	HPLC-MS/MS
TQ24-0204-008	YES. PE-6015	10	Methanol/Water	.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-009	YES.	10	methanol	.	Matrix-matched – Internal standard	HPLC-MS
TQ24-0204-010	-	-	-	-	-	-
TQ24-0204-011	YES.	0	-	Not selected.	Not selected – Not selected	HPLC-MS/MS

FOSETYL-AL (SUM)						
LABORATORY CODE	ACCREDITED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ24-0204-001	-	-	-	-	-	-
TQ24-0204-002	YES.	10	ACN	.	Matrix-matched – Internal standard	HPLC-MS/MS
TQ24-0204-003	YES. FAL_01_B	2	H2O+MeOH	. In House Method	Solvent - Internal standard	HPLC-MS/MS. FAL_01_B
TQ24-0204-004	NO.	5	Acidified MeOH	. QuPPe	Solvent – Internal standard	HPLC-MS/MS
TQ24-0204-005	YES. SOP No.81	10	Methanol	Extraction with solvents.	Matrix-matched – External standard	HPLC-MS/MS
TQ24-0204-006	-	-	-	-	-	-
TQ24-0204-007	YES.	5	methanol	. QuPPe	Matrix-matched – External standard	HPLC-MS/MS
TQ24-0204-008	YES. PE-6015	10	Methanol/Water	.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-009	-	-	-	-	-	-
TQ24-0204-010	-	-	-	-	-	-
TQ24-0204-011	YES.	0		Not selected.	Not selected – Not selected	HPLC-MS/MS

GLYPHOSATE						
LABORATORY CODE	ACCREDITED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ24-0204-001	-	-	-	-	-	-
TQ24-0204-002	-	-	-	-	-	-
TQ24-0204-003	YES. GGA_01_B	2	H2O+MeOH	. In house method	Solvent – Internal standard	HPLC-MS/MS. GGA_01_B
TQ24-0204-004	NO.	5	Acidified MeOH	. QuPPe	Solvent – Internal standard	HPLC-MS/MS
TQ24-0204-005	-	-	-	-	-	-
TQ24-0204-006	-	-	-	-	-	-
TQ24-0204-007	YES.	5	methanol	. QuPPe	Matrix-matched – External standard	HPLC-MS/MS
TQ24-0204-008	YES. PE-681	2	Water	.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-009	YES.	10	methanol	.	Matrix-matched – Internal standard	HPLC-MS/MS
TQ24-0204-010	-	-	-	-	-	-
TQ24-0204-011	YES.	0	-	Not selected.	Not selected – Not selected	HPLC-MS/MS

AMPA						
LABORATORY CODE	ACCREDITED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ24-0204-001	-	-	-	-	-	-
TQ24-0204-002	-	-	-	-	-	-
TQ24-0204-003	YES. GGA_01_B	2	H2O+MeOH	. In house method	Solvent – Internal standard	HPLC-MS/MS. GGA_01_B
TQ24-0204-004	NO.	5	Acidified MeOH	. QuPPe	Solvent – Internal standard	HPLC-MS/MS
TQ24-0204-005	-	-	-	-	-	-
TQ24-0204-006	-	-	-	-	-	-
TQ24-0204-007	-	-	-	-	-	-
TQ24-0204-008	YES. PE-681	2	Water	.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-009	-	-	-	-	-	-
TQ24-0204-010	-	-	-	-	-	-
TQ24-0204-011	-	-	-	-	-	-

CIPROCONAZOLE						
LABORATORY CODE	ACCREDITED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ24-0204-001	YES.	10	ACN	QuEchERS.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-002	YES.	5	ACN	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-003	YES. LMS_01_B	5	H2O+ACN+Acetic Acid	. In House Method	Solvent - Internal standard	HPLC-MS/MS. LMS_01_B
TQ24-0204-004	NO	10	Acetonitrile	Extraction with solvents.	Solvent- Not selected	Other. LC-QTOF
TQ24-0204-005	YES. SOP No.57	10	Acetonitrile	Extraction with solvents.	Matrix-matched – Internal standard	HPLC-MS/MS
TQ24-0204-006	YES.	5	Acetonitrile	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-007	YES.	5	acetonitrile	QuEchERS.	Matrix-matched – External standard	HPLC-MS/MS
TQ24-0204-008	YES. PE-674	10	Acetonitrile	QuEchERS.	Matrix-matched – Method of standard additions	GC MS/MS
TQ24-0204-009	YES.	10	Ethyl acetate	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-010	-	-	-	-	-	-
TQ24-0204-011	-	-	-	-	-	-

CYPRODINIL						
LABORATORY CODE	ACCREDITED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ24-0204-001	YES.	10	ACN	QuEchERS.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-002	NO.	5	ACN	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-003	YES. LMS_01_B	5	H2O+ACN+Acetic Acid	. In House Method	Solvent - Internal standard	HPLC-MS/MS. LMS_01_B
TQ24-0204-004	NO	10	Acetonitrile	Extraction with solvents.	Solvent- Not selected	Other.
TQ24-0204-005	YES. SOP No.57	10	Acetonitrile	Extraction with solvents.	Matrix-matched – Internal standard	GC MS/MS
TQ24-0204-006	-	-	-	-	-	-
TQ24-0204-007	YES.	5	acetonitrile	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-008	YES. PE-674	10	Acetonitrile	QuEchERS.	Matrix-matched – Method of standard additions	GC MS/MS
TQ24-0204-009	YES.	10	Ethyl acetate	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-010	-	-	-	-	-	-
TQ24-0204-011	-	-	-	-	-	-

PENDIMETHALIN						
LABORATORY CODE	ACCREDITED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ24-0204-001	YES.	10	ACN	QuEchERS.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-002	NO.	5	ACN	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-003	YES. GMS_01_B	5	H2O+ACN+Acetic Acid	. In House Method	Solvent - Internal standard	HPLC-MS/MS. GMS_01_B
TQ24-0204-004	NO	10	Acetonitrile	Extraction with solvents.	Solvent- Not selected	Other. LC-QTOF
TQ24-0204-005	YES. SOP No.57	10	Acetonitrile	Extraction with solvents.	Matrix-matched – Internal standard	GC MS/MS
TQ24-0204-006	YES.	5	Acetonitrile	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-007	YES.	5	acetonitrile	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-008	YES. PE-674	10	Acetonitrile	QuEchERS.	Matrix-matched – Method of standard additions	GC MS/MS
TQ24-0204-009	YES.	10	Ethyl acetate	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-010	-	-	-	-	-	-
TQ24-0204-011	-	-	-	-	-	-

IMIDACLOPRID						
LABORATORY CODE	ACCREDITED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ24-0204-001	YES.	10	ACN	QuEchERS.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-002	YES.	5	ACN	QuEchERS.	Matrix-matched – External standard	HPLC-MS/MS
TQ24-0204-003	YES. LMS_01_B	5	H2O+ACN+Acetic Acid	. In House Method	Solvent - Internal standard	HPLC-MS/MS. LMS_01_B
TQ24-0204-004	NO	10	Acetonitrile	Extraction with solvents.	Solvent- Not selected	Other. LC-QTOF
TQ24-0204-005	YES. SOP No.57	10	Acetonitrile	Extraction with solvents.	Matrix-matched – Internal standard	HPLC-MS/MS
TQ24-0204-006	-	-	-	-	-	-
TQ24-0204-007	YES.	5	acetonitrile	QuEchERS.	Matrix-matched – External standard	HPLC-MS/MS
TQ24-0204-008	YES. PE-674	10	Acetonitrile	QuEchERS.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-009	YES.	10	Acetonitrile	QuEchERS.	Matrix-matched – External standard	HPLC-MS/MS
TQ24-0204-010	-	-	-	-	-	-
TQ24-0204-011	-	-	-	-	-	-

PYRIPROXYFEN						
LABORATORY CODE	ACCREDITED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ24-0204-001	YES.	10	ACN	QuEchERS.	Matrix-matched – Method of standard additions	HPLC-MS/MS
TQ24-0204-002	NO.	5	ACN	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-003	YES. GMS_01_B	5	H2O+ACN+Acetic Acid	. In House Method	Solvent - Internal standard	HPLC-MS/MS. GMS_01_B
TQ24-0204-004	NO	10	Acetonitrile	Extraction with solvents.	Solvent- Not selected	Other.
TQ24-0204-005	-	-	-	-	-	-
TQ24-0204-006	YES.	5	Acetonitrile	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-007	YES.	5	acetonitrile	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-008	YES. PE-674	10	Acetonitrile	QuEchERS.	Matrix-matched – Method of standard additions	GC MS/MS
TQ24-0204-009	YES.	10	Ethyl acetate	QuEchERS.	Matrix-matched – External standard	GC MS/MS
TQ24-0204-010	-	-	-	-	-	-
TQ24-0204-011	-	-	-	-	-	-

10. REFERENCES

TestQual Proficiency Testing Schemes are based on the following standards:

- UNE-EN ISO/IEC 17043, into force. Conformity assessment- General requirements for proficiency testing.
- ISO13528 into force. 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/11312/2021 (Implemented by 01/01/2022) Guidance document on analytical quality control and method validation procedures for pesticides residues analysis in food and feed.
- Commission Regulation (EU) 2021/1807 of 13 October 2021 amending Annexes II, III and IV to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for acibenzolar-S-methyl, aqueous extract from the germinated seeds of sweet Lupinus albus, azoxystrobin, clopyralid, cyflufenamid, fludioxonil, fluopyram, **FOSETYL**, metazachlor, oxathiapiprolin, tebufenozide and thiabendazole in or on certain products
  OJ L 16, 23.1.2016, p. 8–20: ES // EN
- Commission Regulation (EU) No 293/2013 of 20 March 2013 amending Annexes II and III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for emamectin benzoate, etofenprox, etoxazole, flutriafol, GLYPHOSATE, phosmet, pyraclostrobin, spinosad and spirotetramat in or on certain products.
  DO L 96 de 5.4.2013, p. 1/30: ES // EN

END OF THE REPORT