

TESTQUAL

PROFICIENCY TESTING SCHEMES

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(Proficiency Testing Schemes)

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FINAL REPORT TestQual 159

Pesticides, including phenoxyacid Pesticides, Fosetyl and Glyphosate in Strawberry

LABORATORY:	AGQ MAROC
LABORATORY CODE:	TQ22-0159-010
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0. GLOSARY AND ABBREVIATIONS

Text	Abbreviation
TestQual	TQ
Proficiency test	PT / P.T.
Limit Of Quantification	LOQ
NA	Not Analysed
RSD _A	Assigned Relative Standard Deviation
FLUAZIFOP-P (SUM OF ALL THE CONSTITUENT ISOMERS OF FLUAZIFOP, ITS ESTERS AND ITS CONJUGATES, EXPRESSED AS FLUAZIFOP)	FLUAZIFOP (SUM)
MCPA AND MCPB (MCPA, MCPB INCLUDING THEIR SALTS, ESTERS AND CONJUGATES EXPRESSED AS MCPA)	MCPA (SUM)
Fosetyl-Al (sum of fosetyl, phosphonic acid and their salts, expressed as fosetyl)	FOSETYL-AL (SUM)

SUMMARY

The samples from this proficiency test were sent on Mayo of 2022 to **34** participant laboratories and **33** sent their results.

Summary TestQual 159- Strawberry results:

ANALYTE	ACCREDITED?	NUMBER OF DATA*	ASSIGNED VALUE (µg/Kg)	UNCERTAINTY (µg/Kg) **	%RSD _A	TARGET STANDARD DEVIATION (µg/Kg)	ROBUST STANDARD DEVIATION (µg/Kg)
CYPRODINIL	YES	26	112.41	2.97	25	28.10	15.16
FENHEXAMID	YES	25	84.21	2.35	25	21.05	11.75
HEXYTHIAZOX	YES	25	284.77	9.67	25	71.19	48.36
MYCLOBUTANIL	YES	25	142.39	3.96	25	35.60	19.82
FLUAZIFOP (SUM)	YES	25	153.15	6.53	25	38.29	32.63
MCPA (SUM)	YES	21	312.94	11.49	25	78.24	52.67
FOSETYL	YES	22	226.27	12.34	25	56.57	57.86
PHOSPHONIC ACID	YES	27	376.79	16.29	25	94.20	84.66
FOSETYL-AL (SUM)	YES	27	712.09	32.44	25	178.02	168.54
GLYPHOSATE	YES	25	130.82	5.03	25	32.71	25.13

*Results considered extreme outliers have not been considered

**Uncertainty NOT negligible.

ANALITE	NUMBER OF Z-SCORES*	% SATISFACTORY	% QUESTIONABLE	% UNSATISFACTORY
CYPRODINIL	26	100	0	0
FENHEXAMID	26	96	4	0
HEXYTHIAZOX	26	96	4	0
MYCLOBUTANIL	26	96	4	0
FLUAZIFOP (SUM)	25	100	0	0
MCPA (SUM)	24	88	0	13
FOSETYL	29	79	3	17
PHOSPHONIC ACID	29	93	3	3
FOSETYL-AL (SUM)	29	93	0	7
GLYPHOSATE	27	93	7	0

**Every result has been assigned with a 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 159 Strawberry** 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 **Pesticides, including phenoxyacid Pesticides, Fosetyl and Glyphosate in Strawberry**. After the evaluation of the applications (depending on the LOQ of the laboratory and the checking the participant will receive the sample correctly), **34** laboratories were accepted, and the test material was sent in **May of 2022**. The assigned concentration value ($\mu\text{g}/\text{kg}$) 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 $|z| \leq 2$, questionable if $2 \leq |z| \leq 3$ and unsatisfactory if $|z| > 3$.

The most important dates of the proficiency test have been:

Date	Activity	Carried out by
09/May & 10/May 2022	Sample delivery	TestQual
10/Jun/2022	Final date to submit results	Participants
17/Jun/2022	Final report (Email and/or client area)	TestQual

Program coordinators: José Pedro Navarro Vicente

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 jpnavarro@testqual.com within the next 60 days after the report has been issued.

2. TEST MATERIAL. DISTRIBUTION AND CONTROL.

TestQual 159 scheme is a proficiency test based in the analysis of **Pesticides, including phenoxyacid Pesticides, Fosetyl and Glyphosate in Strawberry** that has been spiked with the following **standards**:

STANDARD

CYPRODINIL
FENHEXAMID
HEXYTHIAZOX
MYCLOBUTANIL
FLUAZIFOP-P-Butyl
MCPA (acid form)
FOSETYL (MW=109.04 g/mol)
PHOSPHONIC ACID
GLYPHOSATE

About **16 Kg** of matrix was bought in an specialized shop in Spain.

The material was cut in very small pieces, dropped into liquid N₂, 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 lot of samples was ready the sample were stored in a temperature-controlled freezer below -20°C until the dispatch of the samples.

Each sample had approximately **150 g**. Once the lot of samples 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 lot of samples 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 **Pesticides, including phenoxyacid Pesticides, Fosetyl and Glyphosate** 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.

The **assigned value (X)** was determined 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.

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 taken into account for the calculation of the assigned value.

The **standard uncertainty (u_x)** 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.

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 U_x 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:

	$ 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.

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

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

Being $b_i = \%_{\text{RSDA}}/100$, and $\%_{\text{RSDA}}$ 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.

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.

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:

$ Z \leq 2$	Satisfactory
$2 < Z \leq 3$	Questionable
$ Z > 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:

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, 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 (10 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 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:

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

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

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.

5. RESULTS

5.1. RESULTS, LIMITS OF QUANTIFICATION AND Z-SCORE

Legend:

NO:	Analyte no informed (not detected) by the participant (false negative).	X:	Assigned value.
<LOQ:	Analyte in a concentration lower than the participant's limit of quantification.	u _x :	Uncertainty of the assigned value.
*A:	Result considered as statistically aberrant and not considered to calculate the assigned value.	X _i :	Participant's reported value.
Bold	Results with z-score ≥ 2 .	LOQ:	Participant's Limit of quantification.
*1	Uncertainty of the assigned value NOT negligible. Issued evaluation only for informative purposes.	NA:	Analyte not analysed by the participant.
		σ̂:	Target standard deviation

	CYPRODINIL			FENHEXAMID			HEXYTHIAZOX			MYCLOBUTANIL		
	(X= 112.41 μg/Kg)	(Ux= 2.97 μg/Kg)	(σ̂= 28.10 μg/Kg)	(X= 84.21 μg/Kg)	(Ux= 2.35 μg/Kg)	(σ̂= 21.05 μg/Kg)	(X= 284.77 μg/Kg)	(Ux= 9.67 μg/Kg)	(σ̂= 71.19 μg/Kg)	(X= 142.39 μg/Kg)	(Ux= 3.96 μg/Kg)	(σ̂= 35.60 μg/Kg)
LABORATORY CODE	X _i μg/Kg	LOQ μg/Kg	z-score	X _i μg/Kg	LOQ μg/Kg	z-score	X _i μg/Kg	LOQ μg/Kg	z-score	X _i μg/Kg	LOQ μg/Kg	z-score
TQ22-0159-001	100	10	-0.4	81	10	-0.2	310	10	0.4	120	10	-0.6
TQ22-0159-002	105	10	-0.3	93	10	0.4	297	10	0.2	185	10	1.2
TQ22-0159-003	105	10	-0.3	88	10	0.2	293	10	0.1	144	10	0.0
TQ22-0159-004	NA			NA			NA			NA		
TQ22-0159-005	131	10	0.7	90.3	10	0.3	312	10	0.4	158	10	0.4
TQ22-0159-006	NA			NA			NA			NA		
TQ22-0159-007	97	10	-0.5	82	10	-0.1	250	10	-0.5	128	10	-0.4
TQ22-0159-008	NA			NA			NA			NA		

	CYPRODINIL			FENHEXAMID			HEXYTHIAZOX			MYCLOBUTANIL		
	(X= 112.41 µg/Kg)			(X= 84.21 µg/Kg)			(X= 284.77 µg/Kg)			(X= 142.39 µg/Kg)		
	(Ux= 2.97 µg/Kg)			(Ux= 2.35 µg/Kg)			(Ux= 9.67 µg/Kg)			(Ux= 3.96 µg/Kg)		
	$(\hat{\sigma})= 28.10 \mu\text{g/Kg}$			$(\hat{\sigma})= 21.05 \mu\text{g/Kg}$			$(\hat{\sigma})= 71.19 \mu\text{g/Kg}$			$(\hat{\sigma})= 35.60 \mu\text{g/Kg}$		
LABORATORY CODE	X _i µg/Kg	LOQ µg/Kg	z-score	X _i µg/Kg	LOQ µg/Kg	z-score	X _i µg/Kg	LOQ µg/Kg	z-score	X _i µg/Kg	LOQ µg/Kg	z-score
TQ22-0159-009	-	10		-	10		-	10		-	10	
TQ22-0159-010	123	10	0.4	105	10	1.0	290	10	0.1	150	10	0.2
TQ22-0159-011	NA			NA			NA			NA		
TQ22-0159-012	96	10	-0.6	72	10	-0.6	330	10	0.6	120	10	-0.6
TQ22-0159-013	96	10	-0.6	82	10	-0.1	250	10	-0.5	120	10	-0.6
TQ22-0159-014	108	10	-0.2	83	10	-0.1	265	10	-0.3	127	10	-0.4
TQ22-0159-015	109	10	-0.1	95	10	0.5	301	10	0.2	137	10	-0.2
TQ22-0159-016	NA			NA			NA			NA		
TQ22-0159-017	105	10	-0.3	88	10	0.2	292	10	0.1	136	10	-0.2
TQ22-0159-018	NA			NA			NA			NA		
TQ22-0159-019	128	10	0.6	81	10	-0.2	363	10	1.1	164	10	0.6
TQ22-0159-020	113	10	0.0	82	10	-0.1	281	10	-0.1	149	10	0.2
TQ22-0159-021	106	10	-0.2	76	10	-0.4	263	10	-0.3	130	10	-0.3
TQ22-0159-022	NA			NA			NA			NA		
TQ22-0159-023	97	10	-0.5	65	10	-0.9	207	10	-1.1	140	10	-0.1
TQ22-0159-024	114	10	0.1	81.3	10	-0.1	231	10	-0.8	151	10	0.2
TQ22-0159-025	75.5	10	-1.3	91.5	10	0.3	273.5	10	-0.2	97.5	10	-1.3
TQ22-0159-026	154.89	10	1.5	98.46	10	0.7	461.87^{*A}	10	2.5	159.92	10	0.5
TQ22-0159-027	125	10	0.4	94	10	0.5	350	10	0.9	153	10	0.3
TQ22-0159-028	126	10	0.5	95	10	0.5	338	10	0.7	127	10	-0.4
TQ22-0159-029	124	10	0.4	96	10	0.6	342	10	0.8	137	10	-0.2
TQ22-0159-030	NA			NA			NA			NA		
TQ22-0159-031	154	50	1.5	51	10	-1.6	247	10	-0.5	235^{*A}	10	2.6
TQ22-0159-032	125	10	0.4	140^{*A}	10	2.7	309	10	0.3	170	10	0.8
TQ22-0159-033	99.77	10	-0.4	84.05	10	0.0	298.9	10	0.2	188.19	10	1.3
TQ22-0159-034	115	10	0.1	70	10	-0.7	150	10	-1.9	150	10	0.2
TQ22-0159-035	115	10	0.1	45	10	-1.9	214	10	-1.0	132	10	-0.3

	FOSETYL			PHOSPHONIC ACID			FOSETYL-AL (SUM OF FOSETYL, PHOSPHONIC ACID AND THEIR SALTS. EXPRESSED AS FOSETYL)			GLYPHOSATE		
	(\bar{X} = 226.27 $\mu\text{g/Kg}$)			(\bar{X} = 376.79 $\mu\text{g/Kg}$)			(\bar{X} = 712.09 $\mu\text{g/Kg}$)			(\bar{X} = 130.82 $\mu\text{g/Kg}$)		
	($U_{\bar{X}}$ = 12.34 $\mu\text{g/Kg}$)			($U_{\bar{X}}$ = 16.29 $\mu\text{g/Kg}$)			($U_{\bar{X}}$ = 32.44 $\mu\text{g/Kg}$)			($U_{\bar{X}}$ = 5.03 $\mu\text{g/Kg}$)		
	($\hat{\sigma}$ = 56.57 $\mu\text{g/Kg}$)			($\hat{\sigma}$ = 94.20 $\mu\text{g/Kg}$)			($\hat{\sigma}$ = 178.02 $\mu\text{g/Kg}$)			($\hat{\sigma}$ = 32.71 $\mu\text{g/Kg}$)		
LABORATORY CODE	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score
TQ22-0159-001	200	50	-0.5	290	10	-0.9	590	50	-0.7	130	10	0.0
TQ22-0159-002	269	10	0.8	532	10	1.6	982	10	1.5	64	10	-2.0
TQ22-0159-003	120	10	-1.9	361	10	-0.2	600	10	-0.6	105	10	-0.8
TQ22-0159-004	NO	10	-3.9	347	10	-0.3	465	10	-1.4	141	10	0.3
TQ22-0159-005	NA			NA			NA			NA		
TQ22-0159-006	256.2	10	0.5	428	10	0.5	830	10	0.7	157.1	10	0.8
TQ22-0159-007	-	10		-	50		674	10	-0.2	105	10	-0.8
TQ22-0159-008	231	10	0.1	505	10	1.4	907	10	1.1	119	10	-0.4
TQ22-0159-009	-	100		-	1000		-	1000		-	100	
TQ22-0159-010	197	10	-0.5	353	20	-0.3	670	10	-0.2	155	10	0.7
TQ22-0159-011	NA			NA			NA			NA		
TQ22-0159-012	280	50	0.9	390	50	0.1	600	50	-0.6	120	30	-0.3
TQ22-0159-013	110*^A	10	-2.1	280	10	-1.0	520	10	-1.1	NA		
TQ22-0159-014	206	10	-0.4	406	25	0.3	751	25	0.2	NA		
TQ22-0159-015	772*^A	10	9.6	449	10	0.8	1374*^A	10	3.7	208*^A	10	2.4
TQ22-0159-016	NA			NA			NA			NA		
TQ22-0159-017	256	10	0.5	408	10	0.3	790	10	0.4	141	10	0.3
TQ22-0159-018	196	10	-0.5	394	10	0.2	724	10	0.1	77	10	-1.6
TQ22-0159-019	209	10	-0.3	532	10	1.6	921	10	1.2	139	10	0.2
TQ22-0159-020	580*^A	10	6.3	320	10	-0.6	1009	10	1.7	133	10	0.1
TQ22-0159-021	40*^A	10	-3.3	444	50	0.7	680	50	-0.2	170	20	1.2
TQ22-0159-022	190	10	-0.6	415.4		0.4	746	10	0.2	131	10	0.0
TQ22-0159-023	204	10	-0.4	280	50	-1.0	576.4	10	-0.8	130	10	0.0
TQ22-0159-024	NA			NA			NA			NA		
TQ22-0159-025	232	10	0.1	359	10	-0.2	713.06	10	0.0	136.9	10	0.2
TQ22-0159-026	1547*^A	-	23.3	566*^A	1000	2.0	2260*^A	20	8.7	92.4	100	-1.2
TQ22-0159-027	335* ^A	5	1.9	287	5	-1.0	720	10	0.0	106	10	-0.8
TQ22-0159-028	309	5	1.5	307	5	-0.7	720	10	0.0	119	10	-0.4
TQ22-0159-029	314	5	1.6	304	5	-0.8	721	10	0.1	116	10	-0.5

FOSETYL	PHOSPHONIC ACID	FOSETYL-AL (SUM OF FOSETYL, PHOSPHONIC ACID AND THEIR SALTS. EXPRESSED AS FOSETYL)	GLYPHOSATE
(\bar{x} = 226.27 $\mu\text{g/Kg}$)	(\bar{x} = 376.79 $\mu\text{g/Kg}$)	(\bar{x} = 712.09 $\mu\text{g/Kg}$)	(\bar{x} = 130.82 $\mu\text{g/Kg}$)
($U_{\bar{x}}$ = 12.34 $\mu\text{g/Kg}$)	($U_{\bar{x}}$ = 16.29 $\mu\text{g/Kg}$)	($U_{\bar{x}}$ = 32.44 $\mu\text{g/Kg}$)	($U_{\bar{x}}$ = 5.03 $\mu\text{g/Kg}$)
($\hat{\sigma}$ = 56.57 $\mu\text{g/Kg}$)	($\hat{\sigma}$ = 94.20 $\mu\text{g/Kg}$)	($\hat{\sigma}$ = 178.02 $\mu\text{g/Kg}$)	($\hat{\sigma}$ = 32.71 $\mu\text{g/Kg}$)

LABORATORY CODE	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score
TQ22-0159-030	175	20	-0.9	355	25	-0.2	NA			NA		
TQ22-0159-031	205	50	-0.4	NO	10	-3.9	482	50	-1.3	101	100	-0.9
TQ22-0159-032	287	10	1.1	394	10	0.2	815	10	0.6	166	10	1.1
TQ22-0159-033	143.2	10	-1.5	267.1	100	-1.2	501.9	10	-1.2	148.6	10	0.5
TQ22-0159-034	300	10	1.3	500	10	1.3	970	10	1.4	110	10	-0.6
TQ22-0159-035	180	10	-0.8	324	10	-0.6	614	10	-0.6	184	10	1.6

FLUAZIFOP-P (SUM OF ALL THE CONSTITUENT ISOMERS OF FLUAZIFOP. ITS ESTERS AND ITS CONJUGATES. EXPRESSED AS FLUAZIFOP)	MCPA AND MCPB (MCPA, MCPB INCLUDING THEIR SALTS, ESTERS AND CONJUGATES EXPRESSED AS MCPA)
(\bar{x} = 153.15 $\mu\text{g/Kg}$)	(\bar{x} = 312.94 $\mu\text{g/Kg}$)
($U_{\bar{x}}$ = 6.53 $\mu\text{g/Kg}$)	($U_{\bar{x}}$ = 11.49 $\mu\text{g/Kg}$)
($\hat{\sigma}$ = 38.29 $\mu\text{g/Kg}$)	($\hat{\sigma}$ = 78.24 $\mu\text{g/Kg}$)

LABORATORY CODE	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score
TQ22-0159-001	170	10	0.4	220	10	-1.2
TQ22-0159-002	152	10	0.0	NA		
TQ22-0159-003	150	10	-0.1	302	10	-0.1
TQ22-0159-004	81	10	-1.9	248	10	-0.8
TQ22-0159-005	NA			NA		
TQ22-0159-006	NA			NA		
TQ22-0159-007	120	10	-0.9	320	10	0.1
TQ22-0159-008	NA			NA		
TQ22-0159-009	NA			NA		
TQ22-0159-010	NA			NA		
TQ22-0159-011	139	10	-0.4	334	10	0.3
TQ22-0159-012	104	10	-1.3	260	50	-0.7

	FLUAZIFOP-P (SUM OF ALL THE CONSTITUENT ISOMERS OF FLUAZIFOP. ITS ESTERS AND ITS CONJUGATES. EXPRESSED AS FLUAZIFOP)			MCPA AND MCPB (MCPA. MCPB INCLUDING THEIR SALTS. ESTERS AND CONJUGATES EXPRESSED AS MCPA)		
	$(\bar{x}=$	153.15	$\mu\text{g/Kg})$	$(\bar{x}=$	312.94	$\mu\text{g/Kg})$
	$(U\bar{x}=$	6.53	$\mu\text{g/Kg})$	$(U\bar{x}=$	11.49	$\mu\text{g/Kg})$
	$(\hat{\sigma}=$	38.29	$\mu\text{g/Kg})$	$(\hat{\sigma}=$	78.24	$\mu\text{g/Kg})$
LABORATORY CODE	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score	X_i $\mu\text{g/Kg}$	LOQ $\mu\text{g/Kg}$	z-score
TQ22-0159-013	140	10	-0.3	NO	10	-3.9
TQ22-0159-014	222	10	1.8	NO	10	-3.9
TQ22-0159-015	142	10	-0.3	306		-0.1
TQ22-0159-016	NA			NA		
TQ22-0159-017	157	10	0.1	322	10	0.1
TQ22-0159-018	95	10	-1.5	435	10	1.6
TQ22-0159-019	215	10	1.6	NA		
TQ22-0159-020	146	10	-0.2	299	10	-0.2
TQ22-0159-021	175	10	0.6	268	10	-0.6
TQ22-0159-022	NA			NA		
TQ22-0159-023	123	10	-0.8	340	10	0.3
TQ22-0159-024	168	10	0.4	307	10	-0.1
TQ22-0159-025	185.9	10	0.9	591.5*^A	10	3.6
TQ22-0159-026	NA			350.9	10	0.5
TQ22-0159-027	172	10	0.5	351	10	0.5
TQ22-0159-028	155	10	0.0	354	10	0.5
TQ22-0159-029	145	10	-0.2	358	10	0.6
TQ22-0159-030	NA			NA		
TQ22-0159-031	163	10	0.3	322	10	0.1
TQ22-0159-032	NA			NA		
TQ22-0159-033	136.9	10	-0.4	231.68		-1.0
TQ22-0159-034	170	10	0.4	390	10	1.0
TQ22-0159-035	202	10	1.3	280	10	-0.4

5.2. PARTICIPANTS COMMENTS

If any comment was not in English, it has been translated.

Laboratory	Comment
TQ22-0158-001	Fluacifop-P-butyl: 200 ug/kg Fenpropidin: 27 ug/kg.
TQ22-0158-009	This participant informed they would not be able to send results.
TQ22-0158-020	the sample was defrosted when it was received.
TQ22-0158-026	Strawberry PT sample received slightly defrosted (some liquid observed). Package was still cold.

5.3. FALSE POSITIVES AND FALSE NEGATIVES

5.3.1. FALSE POSITIVES:

Laboratory	Analyte	Result/s (µg/Kg)
TQ22-0159-001	Fenpropidin	27

5.3.2. FALSE NEGATIVES:

Laboratory	Analyte (µg/Kg)	LOQ (µg/Kg)	Assigned value (µg/Kg)
TQ22-0158-013	MCPA	10	312.94
TQ22-0158-014	MCPA	10	312.94
TQ22-0158-031	Phosphonic acid	10	376.79

5.4. ASSIGNED VALUE AND TARGET STANDARD DEVIATION

ANALYTE	ACCREDITED?	NUMBER OF DATA*	ASSIGNED VALUE (µg/Kg)	UNCERTAINTY (µg/Kg) **	%RSD _A	TARGET STANDARD DEVIATION (µg/Kg)	ROBUST STANDARD DEVIATION (µg/Kg)
CYPRODINIL	YES	26	112.41	2.97	25	28.10	15.16
FENHEXAMID	YES	25	84.21	2.35	25	21.05	11.75
HEXYTHIAZOX	YES	25	284.77	9.67	25	71.19	48.36
MYCLOBUTANIL	YES	25	142.39	3.96	25	35.60	19.82
FLUAZIFOP (SUM)	YES	25	153.15	6.53	25	38.29	32.63
MCPA (SUM)	YES	21	312.94	11.49	25	78.24	52.67
FOSETYL	YES	22	226.27	12.34	25	56.57	57.86
PHOSPHONIC ACID	YES	27	376.79	16.29	25	94.20	84.66
FOSETYL-AL (SUM)	YES	27	712.09	32.44	25	178.02	168.54
GLYPHOSATE	YES	25	130.82	5.03	25	32.71	25.13

*Results considered extreme outliers have not been considered

**Uncertainty NOT negligible.

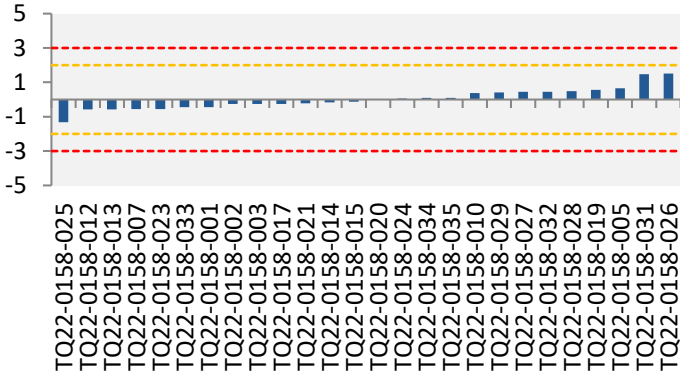
5.5. SATISFACTORY. QUESTIONABLE AND UNSATISFACTORY Z-SCORES

ANALITE	NUMBER OF Z-SCORES*	% SATISFACTORY	% QUESTIONABLE	% UNSATISFACTORY
CYPRODINIL	26	100	0	0
FENHEXAMID	26	96	4	0
HEXYTHIAZOX	26	96	4	0
MYCLOBUTANIL	26	96	4	0
FLUAZIFOP (SUM)	25	100	0	0
MCPA (SUM)	24	88	0	13
FOSETYL	29	79	3	17
PHOSPHONIC ACID	29	93	3	3
FOSETYL-AL (SUM)	29	93	0	7
GLYPHOSATE	27	93	7	0

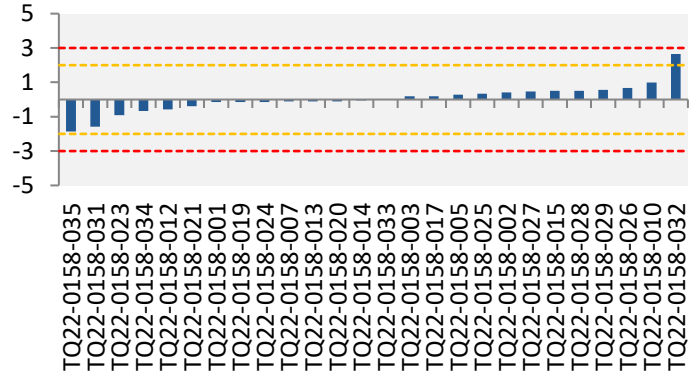
*Every result has been assigned with a z-score, including the results considered as extreme outliers.

6. GRAPHICAL REPRESENTATION OF ASSIGNED Z-SCORES VALUES

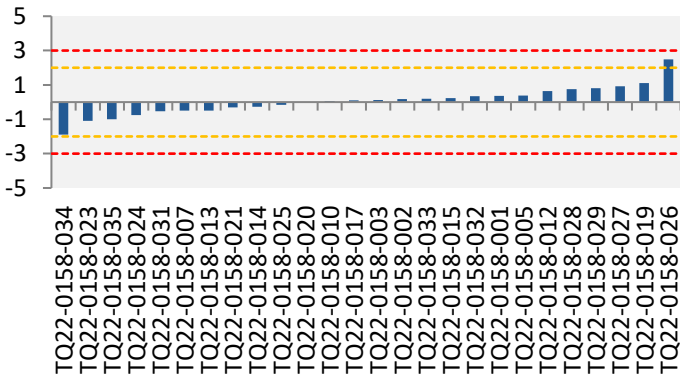
CYPRODINIL



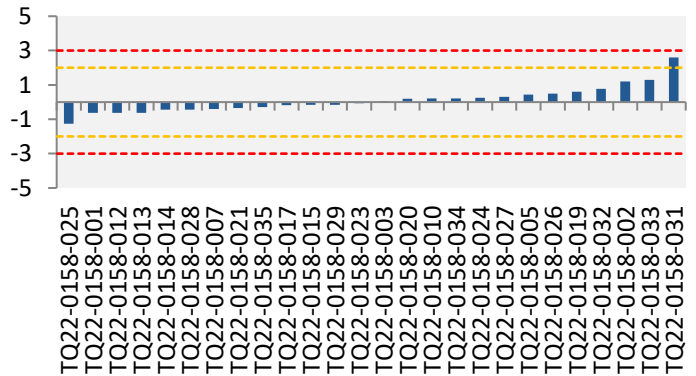
FENHEXAMID



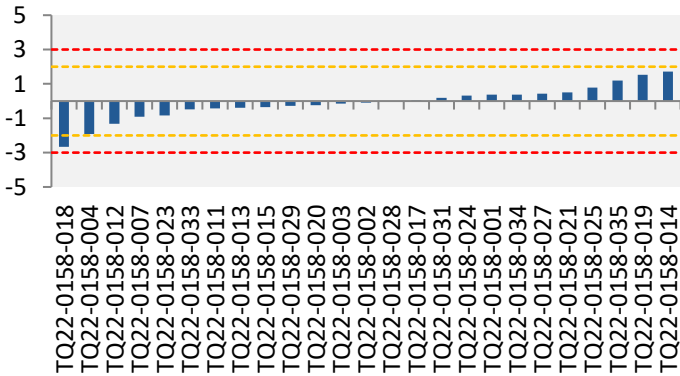
HEXYTHIAZOX



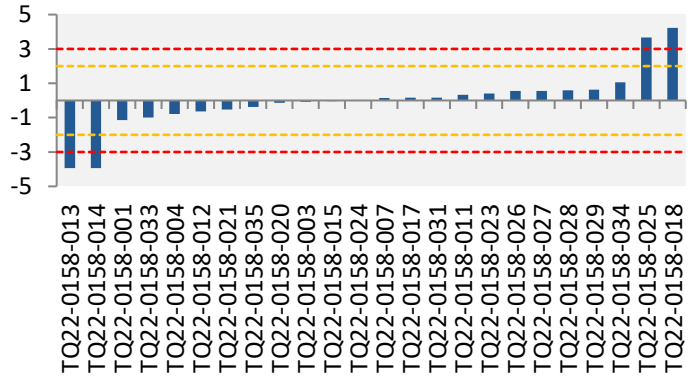
MYCLOBUTANIL



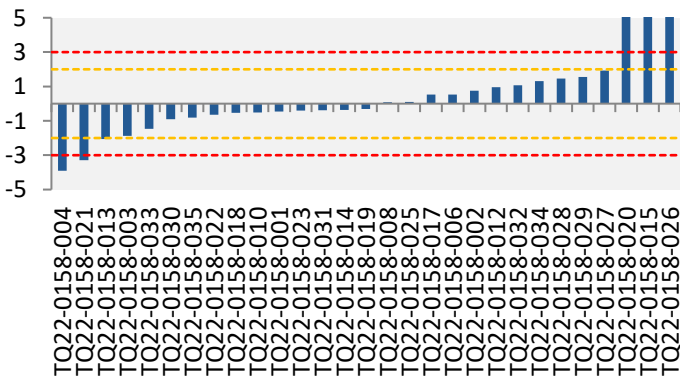
FLUAZIFOP-P (SUM OF ALL THE CONSTITUENT ISOMERS OF FLUAZIFOP, ITS ESTERS AND ITS CONJUGATES, EXPRESSED AS FLUAZIFOP)



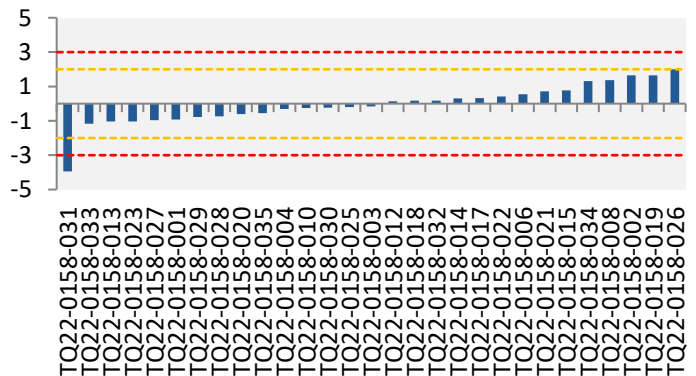
MCPA AND MCPB (MCPA, MCPB INCLUDING THEIR SALTS, ESTERS AND CONJUGATES EXPRESSED AS MCPA)

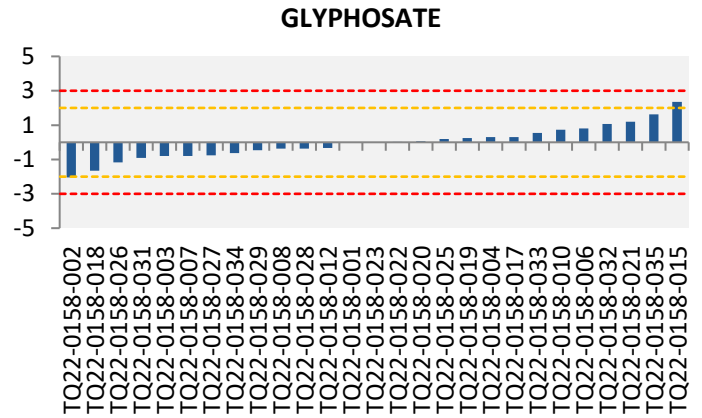
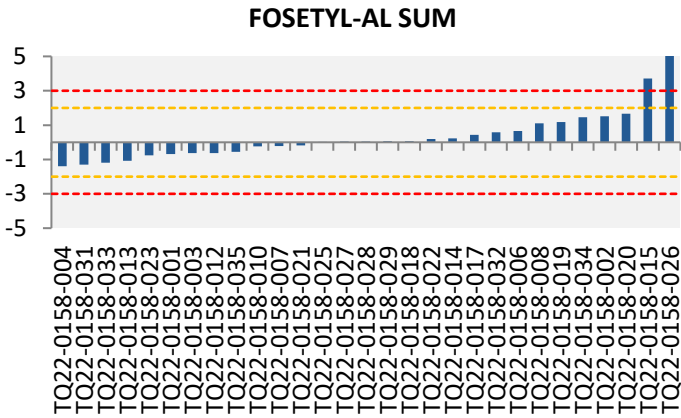


FOSETYL



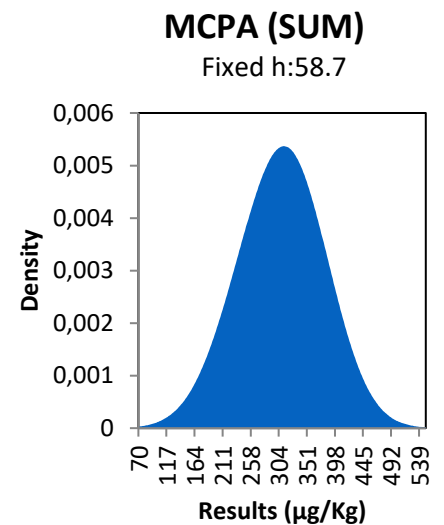
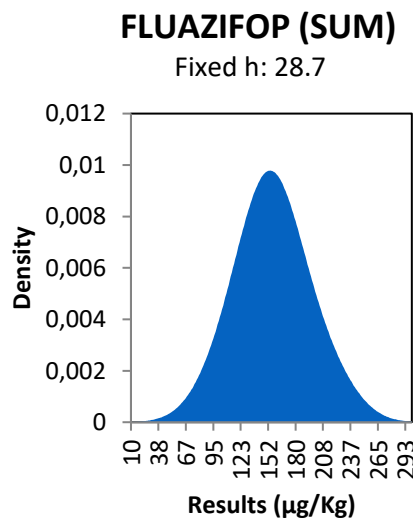
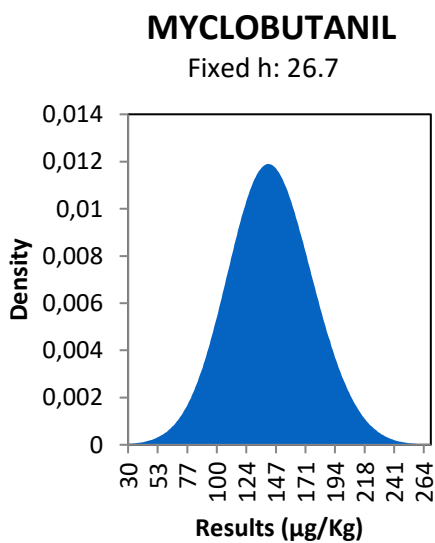
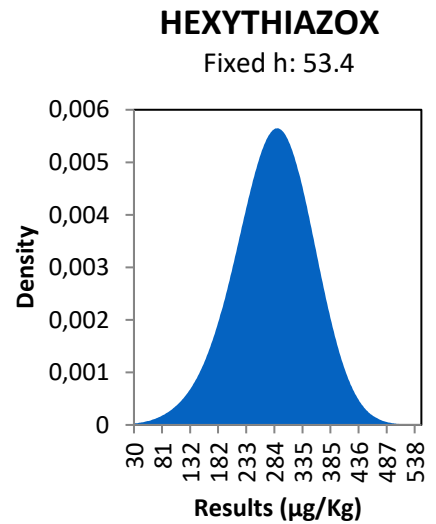
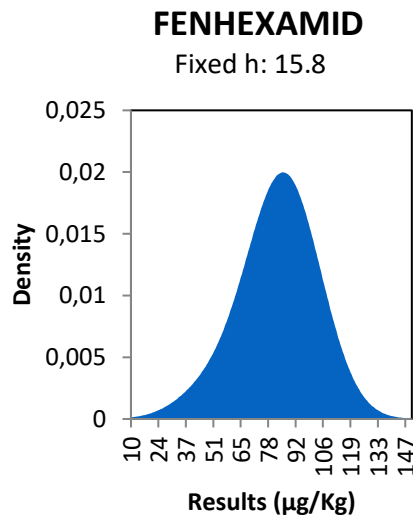
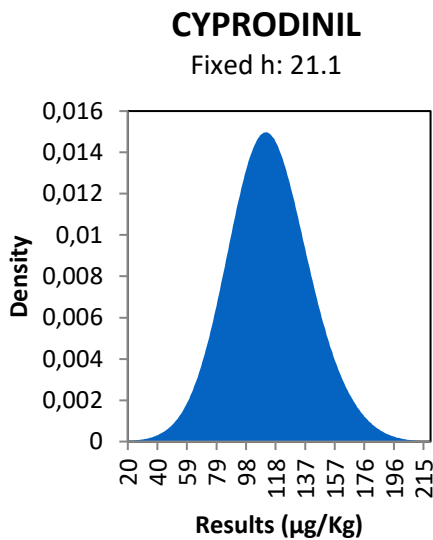
FOSFONIC ACID





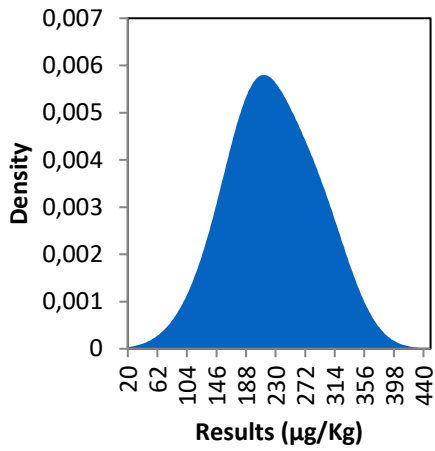
7. RESULTS DISTRIBUTION (KERNEL DENSITY):

For this proficiency test all the analytes have shown an adequate symmetry to be considered unimodal.



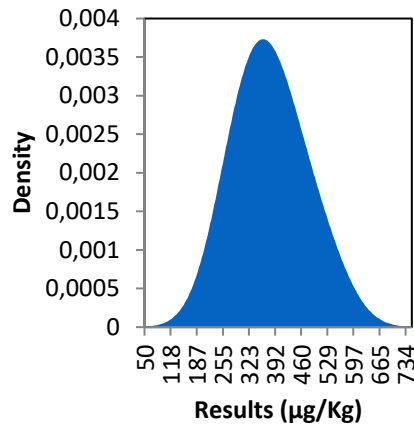
FOSETYL

Fixed h: 42.4



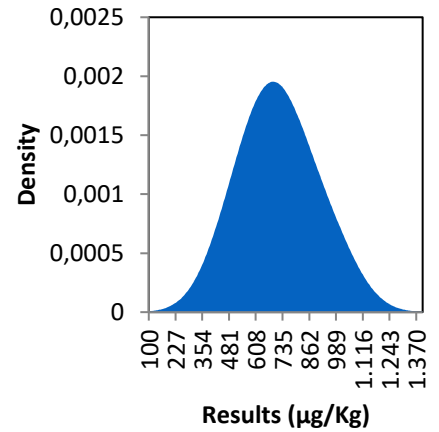
PHOSPHONIC ACID

Fixed h: 70.6



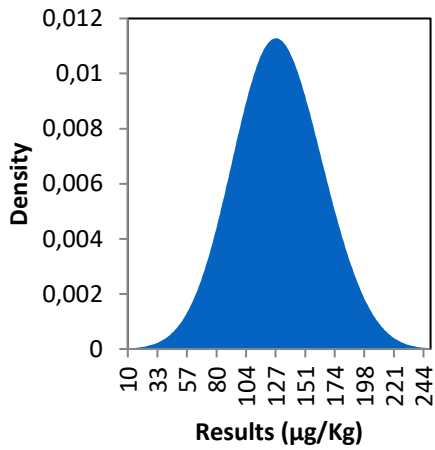
FOSETYL-AL (SUM)

Fixed h: 133.5



GLYPHOSATE

Fixed h: 24.5



8. HOMOGENEITY AND STABILITY OF THE TEST MATERIAL

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

1. The homogeneity check 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 taken into account 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:

HOMOGENEITY	Analyte	CYP ROD INIL	FENHE XAMID	HEXYT HIAZO X	MYCLO BUTAN IL	FLUAZI FOP (SUM)	MCPA (SUM)	FOSET YL	PHOSP HONIC ACID	FOSET YL-AL (SUM)	GLYPHOSATE
		$c < 0.3 \cdot \sigma$	✓	✓	✓	✓	✓	✓	✓	✓	✓
STABILITY	Difference ≤10% t2 vs t1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
	Difference ≤10% t3 vs t1	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓

✓=SATISFIES CRITERIA; X=Does not satisfies criteria

Therefore, the homogeneity and stability of the lot of samples prepared have been satisfactorily checked and passed, meaning that the evaluation do not require these components of the uncertainty to be included in the calculation of the scores to evaluate the performance of the participants.

9. ANALYTICAL METHODS USED BY THE LABORATORIES

Data shown here was sent by participants. If any comment was not in English, it has been translated. Only entries modified are done to keep both the identity and /or nationality confidential (for example by deleting the name of the accreditation body or the language of the ISO). If you require this information displayed, please contact coordinator.

CYPRODINIL						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-001	YES	10	-	QuEchERS	Matrix matched - Internal standard	GC-MS/MS
TQ22-0159-002	-	-	-	-	-	-
TQ22-0159-003	YES	5	ACN/Water	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-004	-	-	-	-	-	-
TQ22-0159-005	YES	10	AcOEt	QuEchERS	Matrix matched - External standard	GC MS/MS.
TQ22-0159-006	-	-	-	-	-	-
TQ22-0159-007	-	-	-	-	-	-
TQ22-0159-008	-	-	-	-	-	-
TQ22-0159-009	-	-	-	-	-	-
TQ22-0159-010	YES	10	AcOEt	QuEchERS	Matrix matched - External standard	GC MS/MS.
TQ22-0159-011	-	-	-	-	-	-
TQ22-0159-012	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-013	YES	10	ACN	QuEchERS	Matrix matched	HPLC-MS/MS.
TQ22-0159-014	NO.	10	ACN	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-015	YES	5	ACN	QuEchERS.	Matrix matched - Internal standard	GC MS/MS.
TQ22-0159-016	-	-	-	-	-	-
TQ22-0159-017	YES	5	Acn	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-018	-	-	-	-	-	-
TQ22-0159-019	-	-	-	-	-	-
TQ22-0159-020	YES	10	ACN	QuEchERS	Matrix matched - External standard	GC MS/MS.
TQ22-0159-021	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	GC MS/MS.

CYPRODINIL						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-022	-	-	-	-	-	-
TQ22-0159-023	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	GC MS/MS.
TQ22-0159-024	NO.	10	ACN	QuEchERS	Matrix matched - External standard	GC MS/MS.
TQ22-0159-025	NO.	10	AcOEt	QuEchERS	Matrix matched - Internal standard	GC MS/MS.
TQ22-0159-026	YES	10	10ml	QuEchERS. Alkaline hydrolysis incl.	Matrix matched - Internal standard	GC MS/MS.
TQ22-0159-027	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-028	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-029	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-030	-	-	-	-	-	-
TQ22-0159-031	YES	10	ACN	QuEchERS	Solvent matched - External standard	GC MS/MS
TQ22-0159-032	YES	10	ACN	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-033	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	LC-MSMS
TQ22-0159-034	YES	10,0	ACN	QuEchERS	Standard addition	LC-MSMS
TQ22-0159-035	YES	15	ACN	QuEchERS	Matrix matched	HPLC-MS/MS.

FENHEXAMID						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-001	YES	10	-	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-002	-	-	-	-	-	-
TQ22-0159-003	YES	5	ACN/Water	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-004	-	-	-	-	-	-
TQ22-0159-005	YES	10	AcOEt	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-006	-	-	-	-	-	-
TQ22-0159-007	-	-	-	-	-	-
TQ22-0159-008	-	-	-	-	-	-

FENHEXAMID							
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE	
TQ22-0159-009	-	-	-	-	-	-	
TQ22-0159-010	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.	
TQ22-0159-011	-	-	-	-	-	-	
TQ22-0159-012	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.	
TQ22-0159-013	YES	10	ACN	QuEchERS	Matrix matched	HPLC-MS/MS.	
TQ22-0159-014	NO.	10	ACN	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS	
TQ22-0159-015	YES	5	ACN	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS	
TQ22-0159-016	-	-	-	-	-	-	
TQ22-0159-017	YES	5	Acn	QuEchERS	Matrix matched - External standard	HPLC-MS/MS	
TQ22-0159-018	-	-	-	-	-	-	
TQ22-0159-019	-	-	-	-	-	-	
TQ22-0159-020	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS	
TQ22-0159-021	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	GC MS/MS.	
TQ22-0159-022	-	-	-	-	-	-	
TQ22-0159-023	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	HPLC-MS/MS	
TQ22-0159-024	NO.	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS	
TQ22-0159-025	NO.	10	ACN	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS	
TQ22-0159-026	YES	10	10ml	QuEchERS. Alkaline hydrolysis incl.	Matrix matched - Internal standard	GC MS/MS.	
TQ22-0159-027	YES	-	-	QuEchERS	Matrix matched	HPLC-MS/MS.	
TQ22-0159-028	YES	-	-	QuEchERS	Matrix matched	HPLC-MS/MS.	
TQ22-0159-029	YES	-	-	QuEchERS	Matrix matched	HPLC-MS/MS.	
TQ22-0159-030	-	-	-	-	-	-	
TQ22-0159-031	YES	10	ACN	QuEchERS	Solvent matched - External standard	GC MS/MS	
TQ22-0159-032	YES	10	ACN	QuEchERS	Matrix matched - Internal standard	GC MS/MS	
TQ22-0159-033	YES	10	ACN	QuEChERS	Solvent matched - Internal standard	LC-MSMS	
TQ22-0159-034	YES	10,0	ACN	QuEChERS	Standard addition	LC-MSMS	
TQ22-0159-035	YES	15	ACN	QuEChERS	Matrix matched	HPLC-MS/MS.	

HEXYTHIAZOX						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-001	YES	10	-	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-002	-	-	-	-	-	-
TQ22-0159-003	YES	5	ACN/Water	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-004	-	-	-	-	-	-
TQ22-0159-005	YES	10	AcOEt	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-006	-	-	-	-	-	-
TQ22-0159-007	-	-	-	-	-	-
TQ22-0159-008	-	-	-	-	-	-
TQ22-0159-009	-	-	-	-	-	-
TQ22-0159-010	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-011	-	-	-	-	-	-
TQ22-0159-012	-	-	-	-	-	-
TQ22-0159-013	YES	10	ACN	QuEchERS	Matrix matched	HPLC-MS/MS.
TQ22-0159-014	NO.	10	ACN	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-015	-	-	-	-	-	-
TQ22-0159-016	-	-	-	-	-	-
TQ22-0159-017	YES	10	Acn	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-018	-	-	-	-	-	-
TQ22-0159-019	-	-	-	-	-	-
TQ22-0159-020	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-021	YES	5	ACN	QuEchERS	Solvent matched - Internal standard	HPLC-MS/MS
TQ22-0159-022	-	-	-	-	-	-
TQ22-0159-023	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	HPLC-MS/MS
TQ22-0159-024	NO.	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-025	NO.	10	ACN	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-026	YES	10	10ml	QuEchERS. Alkaline hydrolysis incl.	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-027	YES	-	-	QuEchERS	Matrix matched	HPLC-MS/MS.

HEXYTHIAZOX						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-028	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-029	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-030	-	-	-	-	-	-
TQ22-0159-031	YES	10	ACN	QuEchERS	Solvent matched - External standard	HPLC-MS/MS.
TQ22-0159-032	YES	10	ACN	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS.
TQ22-0159-033	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	LC-MSMS
TQ22-0159-034	-	-	-	-	-	-
TQ22-0159-035	YES	15	ACN	QuEchERS	Matrix matched	HPLC-MS/MS.

MYCLOBUTANIL						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-001	YES	10	-	QuEchERS	Matrix matched - Internal standard	GC-MS/MS
TQ22-0159-002	-	-	-	-	-	-
TQ22-0159-003	YES	5	ACN/Water	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-004	-	-	-	-	-	-
TQ22-0159-005	YES	10	AcOEt	QuEchERS	Matrix matched - External standard	GC MS/MS.
TQ22-0159-006	-	-	-	-	-	-
TQ22-0159-007	-	-	-	-	-	-
TQ22-0159-008	-	-	-	-	-	-
TQ22-0159-009	-	-	-	-	-	-
TQ22-0159-010	YES	10	AcOEt	QuEchERS	Matrix matched - External standard	GC MS/MS.
TQ22-0159-011	-	-	-	-	-	-
TQ22-0159-012	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-013	YES	10	ACN	QuEchERS	Matrix matched	HPLC-MS/MS.
TQ22-0159-014	NO.	10	ACN	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS

MYCLOBUTANIL						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-015	YES	5	ACN	QuEchERS.	Matrix matched - Internal standard	GC MS/MS.
TQ22-0159-016	-	-	-	-	-	-
TQ22-0159-017	-	-	-	-	-	-
TQ22-0159-018	-	-	-	-	-	-
TQ22-0159-019	-	-	-	-	-	-
TQ22-0159-020	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-021	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	HPLC-MS/MS
TQ22-0159-022	-	-	-	-	-	-
TQ22-0159-023	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	GC MS/MS.
TQ22-0159-024	No.	10	ACN	QuEchERS	Matrix matched - External standard	GC MS/MS.
TQ22-0159-025	YES	10	ACN	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-026	YES	10	10ml	QuEchERS. Alkaline hydrolysis incl.	Matrix matched - Internal standard	GC MS/MS.
TQ22-0159-027	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-028	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-029	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-030	-	-	-	-	-	-
TQ22-0159-031	YES	10	ACN	QuEchERS	Solvent matched - External standard	HPLC-MS/MS.
TQ22-0159-032	YES	10	ACN	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS.
TQ22-0159-033	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	LC-MSMS
TQ22-0159-034	YES	10,0	ACN	QuEchERS	Standard addition	LC-MSMS
TQ22-0159-035	YES	15	ACN	QuEchERS	Matrix matched	HPLC-MS/MS.

FLUAZIFOP (SUM)						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-001	YES	10	-	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-002	-	-	-	-	-	-
TQ22-0159-003	YES	5	ACN/Water	QuEchERS. Hydrolysis	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-004	No	10	-	AH-CB-Quechers (alkaline Hydrolysis)	-	HPLC-MS/MS.
TQ22-0159-005	-	-	-	-	-	-
TQ22-0159-006	-	-	-	-	-	-
TQ22-0159-007	-	-	-	-	-	-
TQ22-0159-008	-	-	-	-	-	-
TQ22-0159-009	-	-	-	-	-	-
TQ22-0159-010	-	-	-	-	-	-
TQ22-0159-011	YES	10	ACN	QuEchERS	Solvent matched - External standard	HPLC-MS/MS.
TQ22-0159-012	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-013	YES	10	ACN	QuEchERS	Matrix matched	HPLC-MS/MS.
TQ22-0159-014	NO.	10	ACN	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-015	YES	5	ACN	QuEchERS. Hydrolysis	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-016	-	-	-	-	-	-
TQ22-0159-017	YES	5	Acn	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-018	-	-	-	-	-	-
TQ22-0159-019	-	-	-	-	-	-
TQ22-0159-020	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-021	YES	5	ACN	QuEchERS	Solvent matched - Internal standard	HPLC-MS/MS
TQ22-0159-022	-	-	-	-	-	-
TQ22-0159-023	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	HPLC-MS/MS
TQ22-0159-024	NO.	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-025	YES	10	ACN	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-026	-	-	-	-	-	-

FLUAZIFOP (SUM)						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-027	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-028	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-029	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-030						
TQ22-0159-031	YES	10	ACN	QuEchERS	Solvent matched - External standard	HPLC-MS/MS.
TQ22-0159-032	-	-	-	-	-	-
TQ22-0159-033	YES	10	ACN	QuEChERS	Solvent matched - Internal standard	LC-MSMS
TQ22-0159-034	YES	10,0	ACN	QuEChERS	Standard addition + Hydrolysis	LC-MSMS
TQ22-0159-035	-	-	-	-	-	-

MCPA (SUM)						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-001	YES	10	-	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-002	-	-	-	-	-	-
TQ22-0159-003	YES	5	ACN/Water	QuEchERS. Hydrolysis	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-004	YES	10	-	AH-CB-Quechers (alkaline Hydrolysis)	-	HPLC-MS/MS.
TQ22-0159-005	-	-	-	-	-	-
TQ22-0159-006	-	-	-	-	-	-
TQ22-0159-007	-	-	-	-	-	-
TQ22-0159-008	-	-	-	-	-	-
TQ22-0159-009	-	-	-	-	-	-
TQ22-0159-010	-	-	-	-	-	-
TQ22-0159-011	YES	10	ACN	QuEchERS	Solvent matched - External standard	HPLC-MS/MS.

MCPA (SUM)						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-012	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-013	-	-	-	-	-	-
TQ22-0159-014	-	-	-	-	-	-
TQ22-0159-015	-	-	-	-	-	-
TQ22-0159-016	-	-	-	-	-	-
TQ22-0159-017	YES	5	Acn	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-018	-	-	-	-	-	-
TQ22-0159-019	-	-	-	-	-	-
TQ22-0159-020	-	-	-	-	-	-
TQ22-0159-021	YES	5	ACN	QuEchERS	Solvent matched - Internal standard	HPLC-MS/MS
TQ22-0159-022	-	-	-	-	-	-
TQ22-0159-023	YES	10	ACN	QuEchERS	Solvent matched - Internal standard	HPLC-MS/MS
TQ22-0159-024	NO.	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-025	YES	10	ACN	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-026	YES	10	10ml	QuEchERS. Alkaline hydrolysis incl.	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-027	YES	-	-	QuEchERS	Matrix matched	HPLC-MS/MS.
TQ22-0159-028	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-029	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-030	-	-	-	-	-	-
TQ22-0159-031	YES	5	ACN	QuEchERS	Solvent matched - External standard	HPLC-MS/MS.
TQ22-0159-032	-	-	-	-	-	-
TQ22-0159-033	YES	10	ACN	QuEChERS	Solvent matched - Internal standard	LC-MSMS
TQ22-0159-034	YES	10,0	ACN	QuEChERS	Standard addition + Hydrolysis	LC-MSMS
TQ22-0159-035	YES	15	ACN	QuEChERS	Matrix matched	HPLC-MS/MS.

FOSETYL AND PHOSPHONIC ACID						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-001	YES	10	-	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-002	YES	10	MeOH	Solvent extraction.	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-003	YES	5	MeOH/Water	QuPPE	Solvent matched - Internal standard	HPLC-MS/MS.
TQ22-0159-004	YES	10	Acidified methanol	QuPPE	-	LC-MSMS
TQ22-0159-005	-	-	-	-	-	-
TQ22-0159-006	YES ISO 17025:2017	3	MEOH + Formic acid + EDTA water	Solvent extraction	Solvent matched - Internal standard	HPLC-MS/MS.
TQ22-0159-007	-	-	-	-	-	-
TQ22-0159-008	-	-	-	-	-	-
TQ22-0159-009	-	-	-	-	-	-
TQ22-0159-010	YES	10	MeOH	-	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-011	-	-	-	-	-	-
TQ22-0159-012	YES	10	MeOH	Solvent extraction	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-013	YES	10	MEOH 1%	Solvent extraction.	Solvent matched - Internal standard	HPLC-MS/MS.
TQ22-0159-014	NO.	10	MeOH	Solvent extraction.	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-015	-	-	-	-	-	-
TQ22-0159-016	-	-	-	-	-	-
TQ22-0159-017	YES	5	MeOH	Solvent extraction.	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-018	YES	10	-	QuPPE	-	HPLC-MS/MS
TQ22-0159-019	YES	10	-	QuPPE	-	HPLC-MS/MS
TQ22-0159-020	YES	10	MeOH:AF	QuPPE	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-021	YES	5	MeOH	Solvent extraction.	Solvent matched - Internal standard	HPLC-MS/MS
TQ22-0159-022	YES	10	Acidified methanol	Solvent extraction.	Solvent matched - External standard	HPLC-MS/MS
TQ22-0159-023	NO.	10	MeOH	Solvent extraction.	Solvent matched - Internal standard	HPLC-MS/MS.
TQ22-0159-024	-	-	-	-	-	-
TQ22-0159-025	YES	10	MeOH	Solid phase extraction	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-026	YES	10	10ml	QuPPE	Matrix matched	HPLC-MS/MS.

FOSETYL AND PHOSPHONIC ACID						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-027	YES	-	-	QuEchERS	Matrix matched	HPLC-MS/MS.
TQ22-0159-028	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-029	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-030	NO.	10	10	Solvent extraction.	Solvent matched - External standard	HPLC-MS/MS.
TQ22-0159-031	-	-	-	-	-	-
TQ22-0159-032	YES	10	ACN	-	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-033	YES	10	ACN	QuEChERS	Solvent matched - Internal standard	LC-MSMS
TQ22-0159-034	YES	10,0	MeOH/Formic acid (5%)	QuPPE	Standard addition + Internal STD	LC-MSMS
TQ22-0159-035	YES	10	MeOH	-	Matrix matched - Internal standard	HPLC-MS/MS.

GLYPHOSATE						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-001	NO	10	-	QuEchERS	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-002	NO	10	MeOH	Solvent extraction.	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-003	YES	5	MeOH/Water	QuPPE	Solvent matched - Internal standard	HPLC-MS/MS.
TQ22-0159-004	YES	10	Acidified methanol	QuPPE	-	LC-MSMS
TQ22-0159-005	-	-	-	-	-	-
TQ22-0159-006	YES ISO 17025:2017	3	MEOH + Formic acid + EDTA water	Solvent extraction	Solvent matched - Internal standard	HPLC-MS/MS.
TQ22-0159-007	-	-	-	-	-	-
TQ22-0159-008	Sí	-	-	-	-	-
TQ22-0159-009	-	-	-	-	-	-
TQ22-0159-010	YES	2	Other	-	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-011	-	-	-	-	-	-

GLYPHOSATE						
LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ22-0159-012	YES	10	ACN	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ22-0159-013	-	-	-	-	-	-
TQ22-0159-014	-	-	-	-	-	-
TQ22-0159-015	-	-	-	-	-	-
TQ22-0159-016	-	-	-	-	-	-
TQ22-0159-017	YES	5	Water	-	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-018	YES	10	-	QuPpe	-	HPLC-MS/MS
TQ22-0159-019	YES	10	-	QuPpe	-	HPLC-MS/MS
TQ22-0159-020	YES	10	MeOH:AF	QuPpe	Matrix matched - External standard	HPLC-MS/MS
TQ22-0159-021	YES	5	MeOH	Solvent extraction.	Solvent matched - Internal standard	HPLC-MS/MS
TQ22-0159-022	YES	10	Acidified methanol	Solvent extraction.	Solvent matched - External standard	HPLC-MS/MS
TQ22-0159-023	YES	10	MeOH	Solvent extraction.	Solvent matched - Internal standard	HPLC-MS/MS.
TQ22-0159-024	-	-	-	-	-	-
TQ22-0159-025	YES	2	Water	Solid phase extraction	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-026	YES	10	10ml	QuPpe	Matrix matched	HPLC-MS/MS.
TQ22-0159-027	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-028	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-029	YES	-	-	Solvent extraction.	Matrix matched	HPLC-MS/MS.
TQ22-0159-030	-	-	-	-	-	-
TQ22-0159-031	YES	5	Acidified methanol	QuPpe	Solvent matched - External standard	HPLC-MS/MS.
TQ22-0159-032	YES	10	ACN	-	Matrix matched - Internal standard	HPLC-MS/MS
TQ22-0159-033	YES	10	ACN	QuEChERS	Solvent matched - Internal standard	LC-MSMS
TQ22-0159-034	NO	10,0	MeOH/Formic acid (5%)	QuPpe	Standard addition + Internal STD	LC-MSMS
TQ22-0159-035	YES	10	MeOH	-	Matrix matched - Internal standard	HPLC-MS/MS.

10. REFERENCES

TestQual Proficiency Testing Schemes are based on the following standards:

- *UNE-EN ISO/IEC 17043:2010 Conformity assessment — General requirements for proficiency testing.*
- *ISO 13528:2015 Statistical methods for use in proficiency testing by interlaboratory comparison.*
- *THE INTERNATIONAL HARMONIZED PROTOCOL FOR THE PROFICIENCY TESTING OF ANALYTICAL CHEMISTRY LABORATORIES (IUPAC Technical Report.)*
- *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.*
- *Commission Regulation (EU) 2016/75 of 21 January 2016 amending Annex III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for FOSETYL in or on certain products*
OJ L 16, 23.1.2016, p. 8–20
- *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

END OF THE REPORT

