

TESTQUAL

PROFICIENCY TESTING SCHEMES

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(Proficiency Testing Schemes)

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FINAL REPORT TestQual 151

Chlorate, perchlorate and QAC

in Green beans

LABORATORY:	AGQ MAROC
LABORATORY CODE:	TQ21-0151-014
ISSUE DATE OF THE REPORT:	03/01/2022

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GLOSARY AND ABBREVIATIONS

Text	Abbreviation
TestQual	TQ
Proficiency test	PT
Limit Of Quantification	LOQ
NA	Not Analysed
RSD _A	Assigned Relative Standard Deviation
QAC	Quaternary Ammonium Compounds
DidecylDimethylAmmonium Chlorides	DDAC
AlkylBenzyldimethylAmmonium Chlorides	BAC

0. SUMMARY

The samples from this proficiency test were sent on Noviembre of 2021 to **27** participant laboratories and 26 sent their results.

Summary TestQual 151- Green beans 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)
CHLORATE	YES	24	128,81	3,63	25	32,20	17,77
PERCHLORATE	YES	22	49,19	2,61	25	12,30	12,23
BAC C12	YES	15	154,34	9,63	25	38,58	37,30
BAC C14	YES	15	126,08	6,67	25	31,52	25,83
DDAC C12	YES	12	140,12	5,14	25	35,03	17,80

**Results considered extreme outliers have not been considered **Uncertainty NOT negligible.*

ANALITE	NUMBER OF Z-SCORES*	% SATISFACTORY	% QUESTIONABLE	% UNSATISFACTORY
CHLORATE	24	100	0	0
PERCHLORATE	24	92	4	4
BAC C12	15	100	0	0
BAC C14	15	93	7	0
DDAC C12	15	80	7	13

**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 151 Green beans** 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 **Chlorate, perchlorate and QAC** in **Green beans**. After the evaluation of the applications (depending on the LOQ of the laboratory and the checking the participant will receive the sample correctly), **27** laboratories were accepted, and the test material was sent in **November of 2021**. 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
12/Nov/2021 (Week 45)	Closing date for applications	Participants
16/Nov/2021 (Week 46)	Sample shipment	TestQual
10/Dec/2021 (Week 49)	Closing date to send results	Participants
30/Dic/2021 (Week 52)	Final report publication	TestQual

Program coordinators: José Pedro Navarro

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.

2. TEST MATERIAL

TestQual 151 scheme is a proficiency test based in the analysis of **Chlorate, perchlorate and QAC in Green beans** that has been spiked with the following **standards**:

STANDARD
CHLORATE
PERCHLORATE
BAC C12
BAC C14
DDAC C12

About **10 Kg** of matrix was bought in an specialized shop in Spain and analysed by a subcontracted laboratory that holds the standard UNE-EN ISO/IEC 17025 into force.

The material 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 **150-200 g**.

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.

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 **Chlorate, perchlorate and QAC** 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 outside the accreditation 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.

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

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

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.

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	≤ 2	Satisfactory
2 <	Z	≤ 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 for chlorate and perchlorate and 20 µg/kg for QAC**). 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 for chlorate and perchlorate and 20 µg/kg for QAC).

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:

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

Firstly Vs is the variance of the sums S_i :

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

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.

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:

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

LAB.CODE	CHLORATE			PERCHLORATE			BAC C12			BAC C14			DDAC C12		
	(X= 128,81 µg/Kg)			(X= 49,19 µg/Kg)			(X= 154,34 µg/Kg)			(X= 126,08 µg/Kg)			(X= 140,12 µg/Kg)		
	(Ux= 3,63 µg/Kg)			(Ux= 2,61 µg/Kg)			(Ux= 9,63 µg/Kg)			(Ux= 6,67 µg/Kg)			(Ux= 5,14 µg/Kg)		
	(σ̂= 32,20 µg/Kg)			(σ̂= 12,30 µg/Kg)			(σ̂= 38,58 µg/Kg)			(σ̂= 31,52 µg/Kg)			(σ̂= 35,03 µg/Kg)		
	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	X _i µg/Kg	LOQ µg/Kg	z- score
TQ21-0151-001	125,6	10	-0,1	55	10	0,5	NA			NA			NA		
TQ21-0151-002	178,2	10	1,5	48,2	10	-0,1	175,0	10	0,5	117,2	10	-0,3	146,7	10	0,2
TQ21-0151-003	NA			53,6	10	0,4	NA			NA			NA		
TQ21-0151-004	NA			NA			NA			NA			NA		
TQ21-0151-005	86,8	10	-1,3	28,2	10	-1,7	106,3	10	-1,2	84	10	-1,3	127,3	10	-0,4
TQ21-0151-006	129,4	10	0,0	NA	10		NA			NA	10		NA	10	
TQ21-0151-007	130	10	0,0	46	10	-0,3	158	20	0,1	122	20	-0,1	141	20	0,0
TQ21-0151-008	123	10	-0,2	42	10	-0,6	NA			NA	10		NA	10	
TQ21-0151-009	139	5	0,3	66	5	1,4	149	-	-0,1	134	5	0,3	164	5	0,7
TQ21-0151-010	110	10	-0,6	42	10	-0,6	190	10	0,9	149	10	0,7	156	10	0,5
TQ21-0151-011	141	10	0,4	61	10	1,0	139	10	-0,4	120	10	-0,2	148	10	0,2
TQ21-0151-012	129	10	0,0	49,1	10	0,0	139		-0,4	140	10	0,4	111	10	-0,8
TQ21-0151-013	115	10	-0,4	41	10	-0,7	190	10	0,9	191	10	2,1	230^{*A}	10	2,6
TQ21-0151-014	145	10	0,5	58	10	0,7	156	10	0,0	120	10	-0,2	132	10	-0,2
TQ21-0151-015	125	10	-0,1	39	10	-0,8	NA			NA			NA		
TQ21-0151-016	120	10	-0,3	43	10	-0,5	NA			NA			NA		
TQ21-0151-017	76	10	-1,6	75	10	2,1	NA			NA			NA		
TQ21-0151-018	72	10	-1,8	199^{*A}	10	12,2	95	10	-1,5	83	10	-1,4	NO	10	-3,9
TQ21-0151-019	164,0	10	1,1	67,5	10	1,5	118,0	10	-0,9	93,0	10	-1,0	116,0	10	-0,7
TQ21-0151-020	143	10	0,4	31	10	-1,5	195	10	1,1	143	10	0,5	142	10	0,1
TQ21-0151-021	139	10	0,3	66	10	1,4	146		-0,2	137	5	0,3	156	5	0,5
TQ21-0151-022	148	10	0,6	44	10	-0,4	NA			NA			NA		
TQ21-0151-023	120	10	-0,3	42	10	-0,6	NA			NA			NA		
TQ21-0151-024	130	10	0,0	46	10	-0,3	145,38	10	-0,2	129,88	10	0,1	NO	10	-3,9
TQ21-0151-025	-	10			10		-	10			10			10	
TQ21-0151-026	122	50	-0,2	63	50	1,1	NA			NA			NA		
TQ21-0151-027	140	10	0,3	48	10	-0,1	210		1,4	146	10	0,6	139	10	0,0

5.2. PARAMETERS OUTSIDE THE ACCREDITATION. JUSTIFICATION

All analytes in this proficiency test are accredited thanks to their compliance with the requirements for the application of the technical annex into force.

5.3. PARTICIPANTS COMMENTS

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

Laboratory	Comment
TQ21-0151-018	DDAC C12 detected <LOQ

5.4. FALSE POSITIVES AND FALSE NEGATIVES

5.4.1. FALSE POSITIVES:

Laboratory	Analyte	Result (µg/Kg)	LOQ (µg/Kg)
TQ21-0151-013	DDAC C10	31	10
TQ21-0151-012	DDAC C10	12.7	10

5.4.2. FALSE NEGATIVES:

Laboratory	Analyte (µg/Kg)	LOQ (µg/Kg)	Assigned value (µg/Kg)
TQ21-0151-018	DDAC C12	10	140,12

5.5. ASSIGNED VALUE AND TARGET STANDARD DEVIATION

ANALYTE	ACCREDITED?	NUMBER OF DATA *	ASSINGED VALUE (µg/Kg)	UNCERTAINTY (µg/Kg) **	%RSD _A	TARGET STANDARD DEVIATION (µg/Kg)	ROBUST STANDARD DEVIATION (µg/Kg)
CHLORATE	YES	24	128,81	3,63	25	32,20	17,77
PERCHLORATE	YES	22	49,19	2,61	25	12,30	12,23
BAC C12	YES	15	154,34	9,63	25	38,58	37,30
BAC C14	YES	15	126,08	6,67	25	31,52	25,83
DDAC C12	YES	12	140,12	5,14	25	35,03	17,80

*Results considered extreme outliers have not been considered

**Uncertainty NOT negligible.

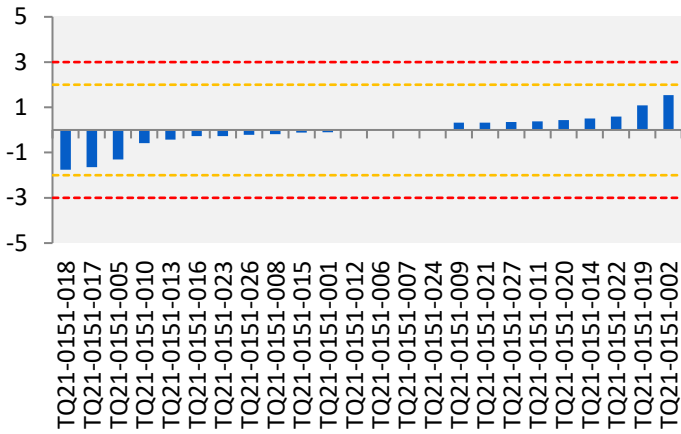
5.6. SATISFACTORY, QUESTIONABLE AND UNSATISFACTORY Z-SCORES

ANALITE	NUMBER OF Z-SCORES*	% SATISFACTORY	% QUESTIONABLE	% UNSATISFACTORY
CHLORATE	24	100	0	0
PERCHLORATE	24	92	4	4
BAC C12	15	100	0	0
BAC C14	15	93	7	0
DDAC C12	15	80	7	13

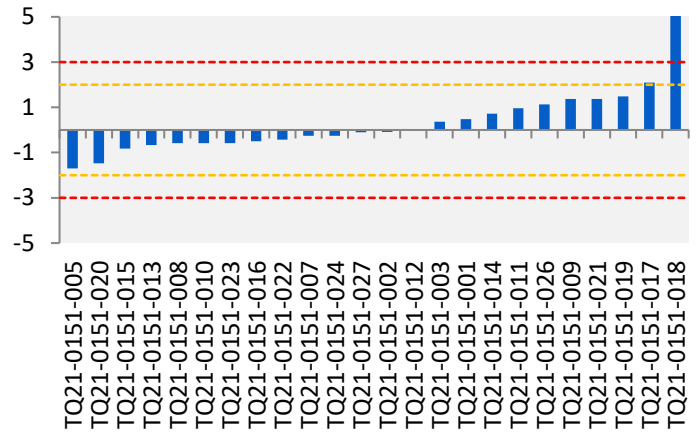
**Every result has been assigned with a z-score, including the results considered as extreme outliers.*

6. GRAPHICAL REPRESENTATION OF ASSIGNED Z-SCORES VALUES

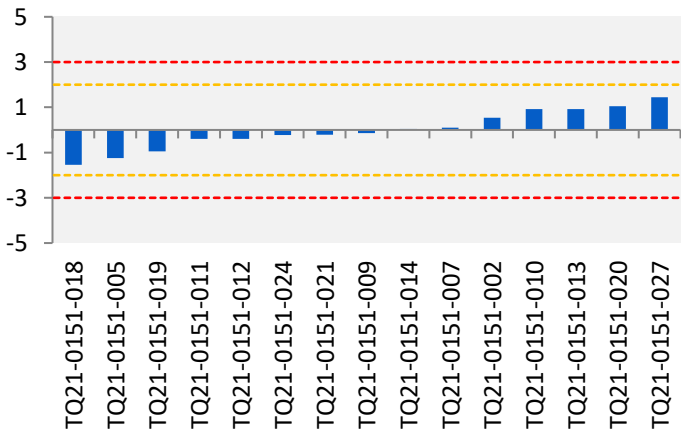
CHLORATE



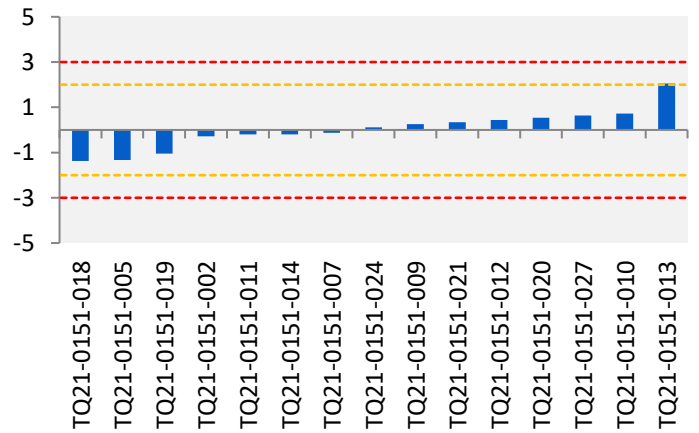
PERCHLORATE



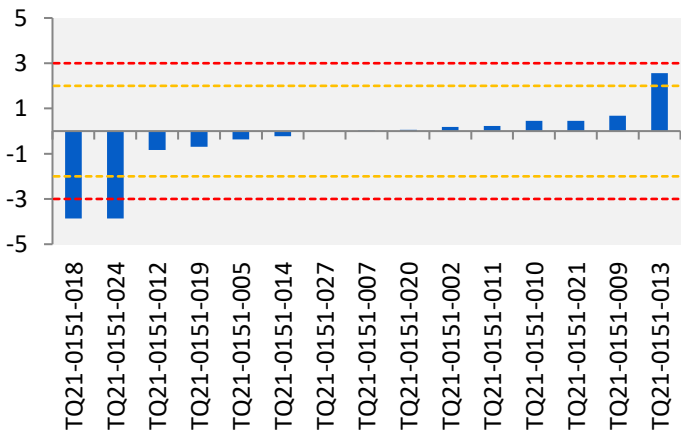
BAC C12



BAC C14



DDAC C12

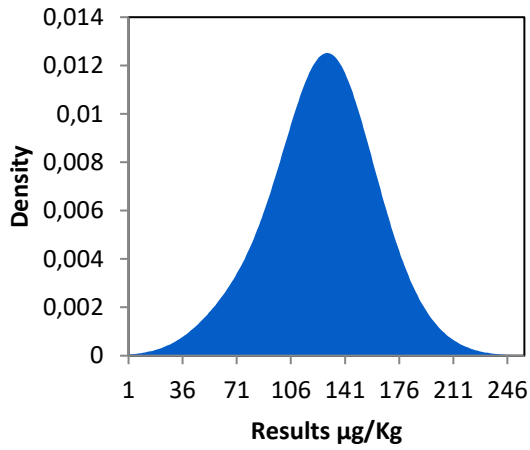


7. RESULTS DISTRIBUTION (KERNEL DENSITY):

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

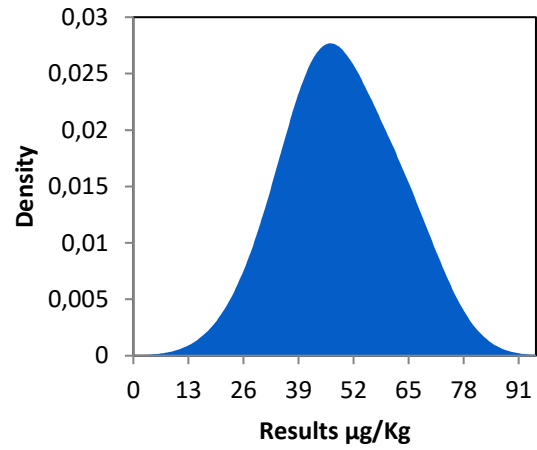
CHLORATE

Fixed h: 24.2



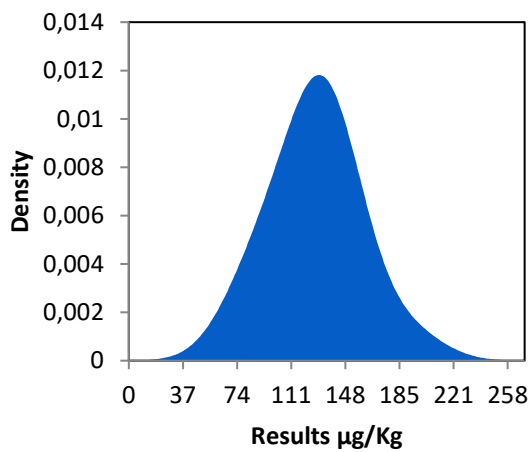
PERCHLORATE

Fixed h: 9.2



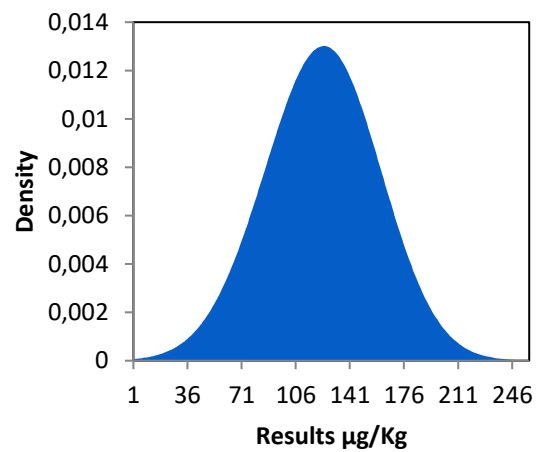
BAC C14

Fixed h: 23.6



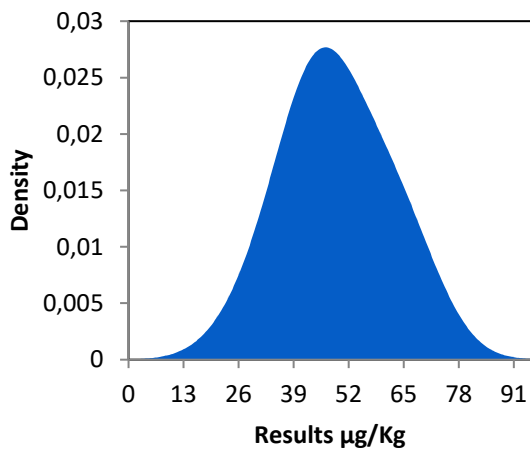
DDAC C12

Fixed h: 26.3



BAC C12

Fixed h: 28.9



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:	Chlorate	Perchlorate	BAC C12	BAC C14	DDAC C12
		$c < 0.3 \cdot \sigma$	Satisfies criteria	Satisfies criteria	Satisfies criteria	Satisfies criteria
STABILITY	$ \text{Difference} \leq 10\%$ $t_2 \text{ vs } t_1$	Satisfies criteria	Satisfies criteria	Satisfies criteria	Satisfies criteria	Satisfies criteria
	$ \text{Difference} \leq 10\%$ $t_3 \text{ vs } t_1$	Satisfies criteria	Satisfies criteria	Satisfies criteria	Satisfies criteria	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 CHLORATE

LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ21-0151-001	YES	-	-	-	-	HPLC-MS/MS.
TQ21-0151-002	YES	-	-	-	-	HPLC-MS/MS.
TQ21-0151-003	-	-	-	-	-	-
TQ21-0151-004	-	-	-	-	-	-
TQ21-0151-005	NO.	10	Methanol	Solvent extraction	Solvent matched	HPLC-MS/MS.
TQ21-0151-006	NO	10	-	Solvent extraction	Solvent matched - External standard	HPLC-MS/MS.
TQ21-0151-007	yes	10	water/MeOH 1% formic acid	solid/liquid Quechers Multipolar	internal standard, solvent matched	HPLC-MS/MS
TQ21-0151-008	yes	10,34	water/MeOH 1% formic acid	solid/liquid Quechers Multipolar	internal standard, solvent matched	HPLC-MS/MS
TQ21-0151-009	yes	2g	Acid methanol	liquid-liquid extraction	matrix matched	LC-MS/MS
TQ21-0151-010	-	-	-	-	-	-
TQ21-0151-011	yes	2g	Acid methanol	liquid-liquid extraction	matrix matched	LC-MS/MS
TQ21-0151-012	YES	10	Methanol acidified	QuPPE	Solvent matched - Internal standard	HPLC-MS/MS.
TQ21-0151-013	YES	10	MeOH 1% Formic acid	QuPPE	Matrix matched - Internal standard	HPLC-MS/MS
TQ21-0151-014	YES IAS	10	METHANOL	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-015	YES	10	Methanol	Solvent extraction	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-016	NO.	10	Methanol	Solvent extractionMethanol	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-017	NO.	5	10 mL 1% HCOOH in methanol	Solvent extraction shaking for 1 min	Solvent matched - External standard	HPLC-MS/MS.
TQ21-0151-018	YES	5	Methanol	-	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-019	NO	10	ACN	Solvent extraction	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-020	YES	10	Methanol	QuEchERS.	Matrix matched	HPLC-MS/MS.
TQ21-0151-021	yes	2g	Acid methanol	liquid-liquid extraction	matrix matched	LC-MS/MS
TQ21-0151-022	NO.	10	methanol acidified con fórmico	QuEchERS.	Matrix matched	HPLC-MS/MS.
TQ21-0151-023	YES	10	-	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS
TQ21-0151-024	YES	-	-	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-025						
TQ21-0151-026	NO.	5	1% formic acid in methanol	Solvent extraction QuPPE Version 12	Matrix matched - External standard	HPLC-MS/MS
TQ21-0151-027	YES	5	ACN	QuPPE	Matrix matched - External standard	HPLC-MS/MS.

PERCHLORATE

LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ21-0151-001	YES	-	-	-	-	HPLC-MS/MS.
TQ21-0151-002	YES	-	-	-	-	HPLC-MS/MS.
TQ21-0151-003	YES	10	methanol	Solvent extraction	Solvent matched - Internal standard	HPLC-MS/MS.
TQ21-0151-004	-	-	-	-	-	-
TQ21-0151-005	NO.	10	Methanol	Solvent extraction	Solvent matched	HPLC-MS/MS.
TQ21-0151-006	-	-	-	-	-	-
TQ21-0151-007	yes	10	water/MeOH 1% formic acid	solid/liquid Quechers Multipolar	internal standard, solvent matched	HPLC-MS/MS
TQ21-0151-008	yes	10,34	water/MeOH 1% formic acid	solid/liquid Quechers Multipolar	internal standard, solvent matched	HPLC-MS/MS
TQ21-0151-009	yes	2g	Acid methanol	liquid-liquid extraction	matrix matched	LC-MS/MS
TQ21-0151-010	-	-	-	-	-	-
TQ21-0151-011	yes	2g	Acid methanol	liquid-liquid extraction	matrix matched	LC-MS/MS
TQ21-0151-012	YES	10	Methanol acidified	QuPpe	Solvent matched - Internal standard	HPLC-MS/MS.
TQ21-0151-013	YES	10	MeOH 1% Formic acid	QuPpe	Matrix matched - Internal standard	HPLC-MS/MS
TQ21-0151-014	YES IAS	10	METHANOL	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-015	YES	10	Methanol	Solvent extraction	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-016	NO.	10	Methanol	Solvent extractionMethanol	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-017	-	-	-	-	-	-
TQ21-0151-018	YES	5	Methanol	-	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-019	NO	10	ACN	Solvent extraction	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-020	YES	10	Methanol	QuEchERS.	Matrix matched	HPLC-MS/MS.
TQ21-0151-021	yes	2g	Acid methanol	liquid-liquid extraction	matrix matched	LC-MS/MS
TQ21-0151-022	NO.	10	methanol acidified con fórmico	QuEchERS.	Matrix matched	HPLC-MS/MS.
TQ21-0151-023	YES	10	-	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS
TQ21-0151-024	YES	-	-	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-025						
TQ21-0151-026	NO.	5	1% formic acid in methanol	Solvent extraction QuPpe Version 12	Matrix matched - External standard	HPLC-MS/MS
TQ21-0151-027	YES	5	ACN	QuPpe	Matrix matched - External standard	HPLC-MS/MS.

BAC C12

LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ21-0151-001	YES	-	-	-	-	HPLC-MS/MS.
TQ21-0151-002	YES	-	-	-	-	HPLC-MS/MS.
TQ21-0151-003	-	-	-	-	-	-
TQ21-0151-004	-	-	-	-	-	-
TQ21-0151-005	NO.	10	ACN	QuEchERS.	Matrix matched	HPLC-MS/MS.
TQ21-0151-006	-	-	-	-	-	-
TQ21-0151-007	yes	10	-	-	-	LC-MS/MS
TQ21-0151-008	-	-	-	-	-	-
TQ21-0151-009	yes	2g	-	QuEChERS	matrix matched	LC-MS/MS
TQ21-0151-010	-	-	-	-	-	-
TQ21-0151-011	yes	2g	-	QuEChERS	matrix matched	LC-MS/MS
TQ21-0151-012	NO.	10	acetonitrile	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ21-0151-013	YES	15	MeCN 1% Acetic acid	QuEchERS.	Solvent matched - External standard	Other. LC-qTOF
TQ21-0151-014	YES IAS	10	ACN	QuEchERS.	Matrix matched - External standard	HPLC-MS/MS.
TQ21-0151-015	-	-	-	-	-	-
TQ21-0151-016	-	-	-	-	-	-
TQ21-0151-017	-	-	-	-	-	-
TQ21-0151-018	YES	5	acetonitrile	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-019	YES	10	acetonitrile	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-020	YES	10	Methanol	QuEchERS.	Matrix matched	HPLC-MS/MS.
TQ21-0151-021	yes	2g	-	QuEChERS	matrix matched	LC-MS/MS
TQ21-0151-022	-	-	-	-	-	-
TQ21-0151-023	-	-	-	-	-	-
TQ21-0151-024	NO	10	ACN	QuEchERS.	Matrix matched	LC-MS/MS
TQ21-0151-025						
TQ21-0151-026	-	-	-	-	-	-
TQ21-0151-027	YES	5	ACN	Matrix matched - External standard	Matrix matched - External standard	HPLC-MS/MS.

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LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ21-0151-001	-	-	-	-	-	-
TQ21-0151-002	YES	-	-	-	-	HPLC-MS/MS.
TQ21-0151-003	-	-	-	-	-	-
TQ21-0151-004	-	-	-	-	-	-
TQ21-0151-005	NO.	10	ACN	QuEchERS.	Matrix matched	HPLC-MS/MS.
TQ21-0151-006	-	-	-	-	-	-
TQ21-0151-007	yes	10	-	-	-	LC-MS/MS
TQ21-0151-008	-	-	-	-	-	-
TQ21-0151-009	yes	2g	-	QuEChERS	matrix matched	LC-MS/MS
TQ21-0151-010	-	-	-	-	-	-
TQ21-0151-011	yes	2g	-	QuEChERS	matrix matched	LC-MS/MS
TQ21-0151-012	NO.	10	acetonitrile	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ21-0151-013	YES	15	MeCN 1% Acetic acid	QuEchERS.	Solvent matched - External standard	Other. LC-qTOF
TQ21-0151-014	YES IAS	10	ACN	QuEchERS.	Matrix matched - External standard	HPLC-MS/MS.
TQ21-0151-015	-	-	-	-	-	-
TQ21-0151-016	-	-	-	-	-	-
TQ21-0151-017	-	-	-	-	-	-
TQ21-0151-018	YES	5	acetonitrile	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-019	YES	10	acetonitrile	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-020	YES	10	Methanol	QuEchERS.	Matrix matched	HPLC-MS/MS.
TQ21-0151-021	yes	2g	-	QuEChERS	matrix matched	LC-MS/MS
TQ21-0151-022	-	-	-	-	-	-
TQ21-0151-023	-	-	-	-	-	-
TQ21-0151-024	NO	10	ACN	QuEchERS.	Matrix matched	LC-MS/MS
TQ21-0151-025						
TQ21-0151-026	-	-	-	-	-	-
TQ21-0151-027	YES	5	ACN	Matrix matched - External standard	Matrix matched - External standard	HPLC-MS/MS.

DDAC C12

LABORATORY CODE	ACREDITATED METHOD?	WEIGHT (g)	EXTRACTION SOLVENT	EXTRACTION TECHNIQUE	CALIBRATION	ANALYSIS TECHNIQUE
TQ21-0151-001	-	-	-	-	-	-
TQ21-0151-002	YES	-	-	-	-	HPLC-MS/MS.
TQ21-0151-003	-	-	-	-	-	-
TQ21-0151-004	-	-	-	-	-	-
TQ21-0151-005	NO.	10	ACN	QuEchERS.	Matrix matched	HPLC-MS/MS.
TQ21-0151-006	-	-	-	-	-	-
TQ21-0151-007	Sí	10	-	-	-	LC-MS/MS
TQ21-0151-008	-	-	-	-	-	-
TQ21-0151-009	yes	2g	-	QuEchERS	matrix matched	LC-MS/MS
TQ21-0151-010	-	-	-	-	-	-
TQ21-0151-011	yes	2g	-	QuEchERS	matrix matched	LC-MS/MS
TQ21-0151-012	NO.	10	acetonitrile	QuEchERS	Matrix matched - External standard	HPLC-MS/MS.
TQ21-0151-013	YES	15	MeCN 1% Acetic acid	QuEchERS.	Solvent matched - External standard	Other. LC-qTOF
TQ21-0151-014	YES IAS	10	ACN	QuEchERS.	Matrix matched - External standard	HPLC-MS/MS.
TQ21-0151-015	-	-	-	-	-	-
TQ21-0151-016	-	-	-	-	-	-
TQ21-0151-017	-	-	-	-	-	-
TQ21-0151-018	-	-	-	-	-	-
TQ21-0151-019	YES	10	acetonitrile	QuEchERS.	Matrix matched - Internal standard	HPLC-MS/MS.
TQ21-0151-020	YES	10	Methanol	QuEchERS.	Matrix matched	HPLC-MS/MS.
TQ21-0151-021	yes	2g	-	QuEchERS	matrix matched	LC-MS/MS
TQ21-0151-022	-	-	-	-	-	-
TQ21-0151-023	-	-	-	-	-	-
TQ21-0151-024	NO	10	ACN	QuEchERS.	Matrix matched	LC-MS/MS
TQ21-0151-025						
TQ21-0151-026	-	-	-	-	-	-
TQ21-0151-027	YES	5	ACN	QuEchERS.	Matrix matched - External 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.)*
- Commission Regulation (EU) No 1119/2014 of 16 October 2014 amending Annex III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for **BENZALKONIUM CHLORIDE** and **DIDECYLDIMETHYLAMMONIUM CHLORIDE** in or on certain products
[OJ L 304, 23.10.2014, p. 43–74](#)
- COMMISSION REGULATION (EU) 2020/749 of 4 June 2020 amending Annex III to Regulation (EC) No 396/2005 of the European Parliament and of the Council as regards maximum residue levels for **CHLORATE** in or on certain products
[OJ L 178, 8.6.2020, p. 7–20](#)
- COMMISSION REGULATION (EU) 2020/685 of 20 May 2020 amending Regulation (EC) No 1881/2006 as regards maximum levels of **PERCHLORATE** in certain foods
[OJ L 160, 25.5.2020, p. 3–5](#)

END OF THE REPORT

