



FOCUS ON THE INTERLABORATORY COMPARISONS REPORT

HOW TO READ THE REPORT ?

Document valide au 11/02/2021

This booklet is made to guide you in understanding and using your first interlaboratory comparisons report (RCIL). For any additional questions, we invite you to contact our sales department:

sales@bipea.org
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WELCOME TO BIPEA!

In a few days you will receive your first samples for analysis. As soon as you receive the samples, please connect you to your member area (on our website www.bipea.org), with your codes and passwords that have been sent to you previously, in order to:

- be aware of the deadlines,
- download and fill out the answer forms concerning your test results,
- obtain a transmission certificate, proof of sending your results to Bipea.

Then, the interlaboratory comparisons report, in French and in English, will be published *.

We have elaborated this booklet in order to help you to understand your first interlaboratory comparisons report. Our PT reports are designed according to the requirements of the paragraph 4.8 from the standard **"ISO/IEC 17043 - Conformity assessment - General requirements for proficiency testing"**. This section describes the elements that have to be included in the report.

This booklet is structured as well as the interlaboratory comparisons report:

- IDENTIFICATION OF THE COMPARISON
- DESCRIPTION OF THE COMPARISON
- RESULTS AND PROFICIENCY ASSESSMENT OF LABORATORIES
- ANNEXES
- ESTIMATION OF ASSIGNED AND TOLERANCE VALUES
- SPECIFICATIONS OF SOME PROGRAMS.

The statistical treatment is realized by our statistical department according to the ISO 13528 standard **"Statistical methods for use in proficiency testing by interlaboratory comparisons"**

** As part of our certification, we assure to publish the report within 17 working days after the deadline of results submission*

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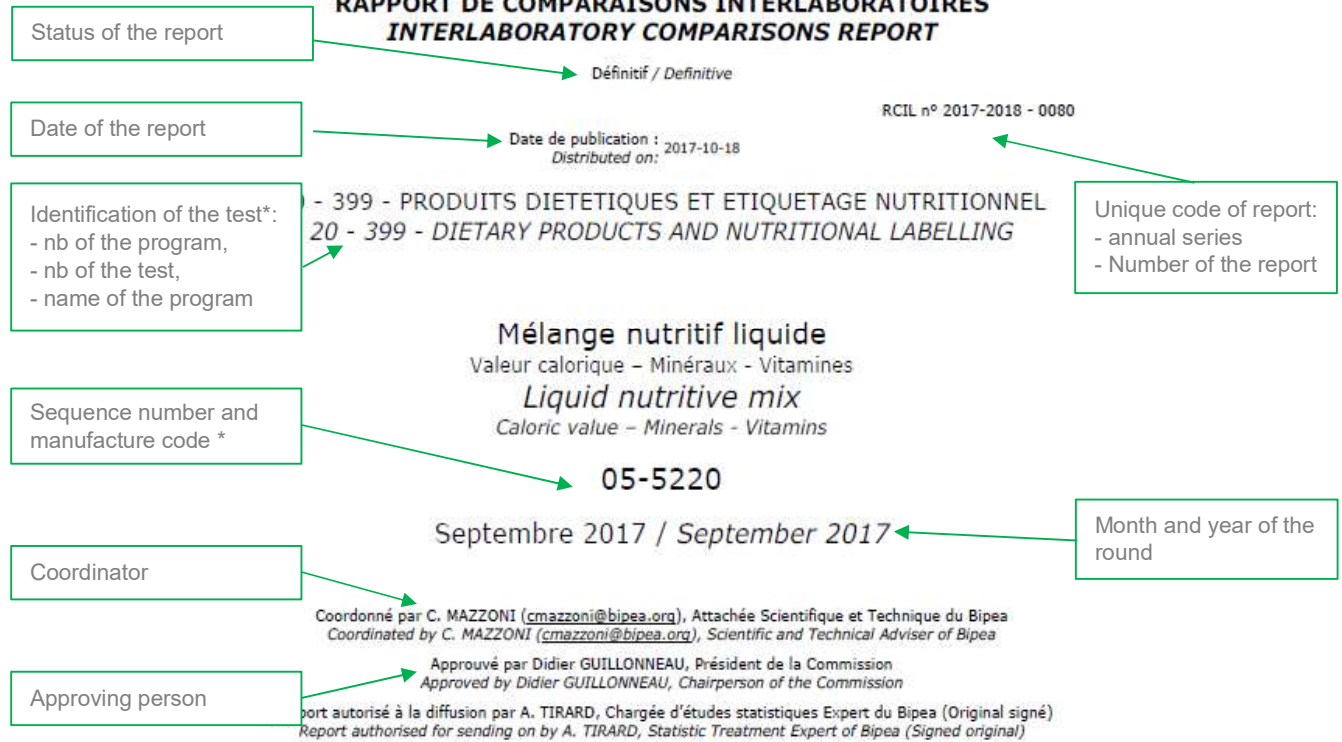
IDENTIFICATION OF THE COMPARAISON

This section gives general information about the comparison (name of the program, matrices, sample code, accreditation of the scheme ...)

* BIPEA internal code



RAPPORT DE COMPARAISONS INTERLABORATOIRES INTERLABORATORY COMPARISONS REPORT



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DESCRIPTION OF THE COMPARAISON

This part provides information about the management and the organization of the proficiency test. Thus it informs about the sample and its manufacture, the homogeneity control, the stability of samples, the statistical treatment...

There is also the comment of the Chairperson Commission and / or BIPEA comment.

1/ CONCEPTION AND ORGANIZATION OF THE PROFICIENCY TESTING SCHEMES

Bipea creates and organizes proficiency testing schemes under annual series which consist of one or several tests. The participants to the scheme choose a series' plan and a statistical plan, during the commission meeting, under scientific and technical department coordination. During the series, the scientific and technical department coordinates the tests with the help of the Chairperson of the commission, who is elected, and his technical group.

2/ PROFICIENCY TEST ITEMS

Product

The product analyzed in September 2017 is: Liquor (Approximative alcoholic strength = 24.10% Vol).

Production

The samples were taken from a provider according to specifications defined by Bipea.

Homogeneity check of the samples

For this test, homogeneity between the samples was verified by experimental studies. The principle of this study is the quantification of the heterogeneity between at least 10 samples in their final packaging, analysed in a random order, by determination of multidimensional homogeneity parameters (spectrum comparisons in SPIR). The results of the control for this test, performed by Bipea by near infrared spectroscopy, have shown that the samples are homogenous enough to meet the requirements of the test.

The conclusions of this study were confirmed by the examination of the robust standard deviations of the laboratories results, which are at the same level as the ones usually observed in similar tests.

Stability check of the samples

For this PTS, experimental stability studies were conducted on samples of rum, brandy, cognac, tequila and mix of spirits for the true alcoholic strength parameter. The analyses were performed either internally by Near Infrared spectroscopy (rum, brandy and cognac) or by an accredited subcontracting laboratory (tequila and mix of spirits). These studies, performed according to the ISO 13528 standard, demonstrated the stability of the samples for the duration of the test.

Moreover, the examination of participants' results shows consistency with previous tests on similar products and confirms the results of the experimental studies.

3/ STATISTICAL TREATMENT

The statistical treatment has been carried out according to the ISO 13528 standard. "Statistical methods for use in proficiency testing by interlaboratory comparisons".

The way to estimate the assigned value and the tolerance value is defined and approved by the participants to the commission meetings and is shown in the table "Assigned value and tolerance values of the specialized commission" available in annex parts.

Just before statistic treatment, data examination is performed according to the following criteria:

- traceability of the provided result (checking of the sample identification number),
- visual (expression of the result, data input error),
- technical (according to the Commission instructions),
- and/or statistic (tests, observed distributions).

Determination of the assigned value

The assigned value or "conventionally true value" (x_{ref}) is:

- determined before the test,
 - using values which come from the formulation,
 - using a value which comes from measurements on national standard device for which metrological traceability is established,
- estimated at the end of the test, using consensus values which come:
 - either from the results of a group of well-known and controlled participating laboratories (the selection criteria are determined by the specialized Commission),
 - either from the results of all the participating laboratories.

Most of the time, the estimator used is the mean obtained from the application of robust algorithm A of ISO 13528 standard. It is given with its standard uncertainty which can quantify the confidence to have on it. However, for specific cases, an adaptation is made according to the nature of the values and the experiment design.

Important note: If the assigned value comes from statistical methods, its reliability is, among other components, directly connected to the number of results which participated in its estimation. Thus, if less than eight results are used to estimate the assigned value, this one is given for information purpose only. The judgment of trueness is then to be interpreted with caution considering the low number of results.

Determination of the tolerance value

The judgment of the trueness of a result is performed according to a tolerance value (VT):

- determined before the test. It could be:
 - a prescription value or a regulatory value,
 - a performance aim of the participants,
 - a value from the reproducibility standard deviation of the standardized analytical method,
 - a value from the standard deviation of the results of the participants during a round of a proficiency test.
- estimated at the end of the test, using the observed dispersion:
 - either from the results of a group of well-known and controlled participating laboratories (the selection criteria are determined by the specialized Commission),
 - either from the results of all the participating laboratories.

The estimate used is generally twice the standard deviation from the robust algorithm A of the ISO 13528 standard.

Calculation of performance statistics

To evaluate proficiency assessment of laboratories, the tolerance value is used to determine a tolerance interval around the assigned value. Outside of this interval, a result will be considered as untrue. The interpretation of the laboratory bias can't be separated from the choice of the tolerance value and from the way to estimate the assigned value.

The performance of the analysis result is indicated under two forms:

- by a visual identification, simultaneously by underlining and attributing a specific color to the value. It corresponds to the estimation of the laboratory measurement error. If this error, which can be positive or negative, is higher, in absolute value, than the tolerance value decided by the commission, it is a signal for the laboratory. This mark requires a detailed examination of the results of the test, via z-score (see below) and/or the construction of accuracy monitoring charts. These complementary tools can help to support the explanations provided for the causes research of the deviation.
- by calculation of a statistic, expressed in score z. The z-score is calculated by taking into account the assigned value and half of the tolerance value. Its absolute value higher than 2.0 is equivalent to a warning signal; an absolute value higher than 3.0 is considered as an action signal.

Remark: There is a correspondence between an underlined result and a z-score which absolute value is more than 2.0, except that a rounding difference. However, in case of a difference due to a mathematical rounding, only the application of the tolerance value by underlining is valid.

Each participant can appeal against the evaluation of its performance by email to the following address: statistiques@bipea.org.

Chairperson comment

IBUTA18: The three highest values were not taken into account for the estimation of the assigned value. They probably correspond to the integration of a peak positioned between isobutanol and formate (Figure 1, page 8).

FORM18: Presence of two populations of results, the first one with 6 results lower than 5 mg.l⁻¹, and a second one with 5 results higher than 5 mg.l⁻¹. The assigned value is given for information purpose and corresponds to the first population. The second one probably corresponds to the integration of an interfering peak, which may be methyl acetate (figure 1, page 8).

The percentage of underlined results is quite important, close to 20% and more, for:

- Ethyl acetate (ACET18) and esters GC as ethyl acetate (CPGE), influenced by the result of ethyl acetate,
- Hexanol (HEXA18) despite a quite high abundance in this sample,
- Ethyl laurate (LAURA18), sum of volatile substances (SSVOL, with still results expressed as mg.l⁻¹ instead of g.hL⁻¹ PA) and Copper (Cu18),
- 3-cis 1-hexenol (CHEX18), with a low assigned value and a tail of distribution. Higher results probably correspond to the presence of an interfering compound (Figure 2, page 9).

Guillaume SNAKKERS, Chairperson of the Commission

RESULTS & PROFICIENCY ASSESSMENT OF LABORATORIES

This part details the results of the participating laboratories by using tables, histograms and also graphs issued from the statistical treatment, that permit to evaluate the proficiency assessment of laboratories.

- Results tables and proficiency assessment

The results tables are composed by two parts: a first one details the estimated statistical parameters and a second one shows the laboratory results. The analytical criteria are shown in columns while the laboratories edition codes are shown in lines.

Those tables show the proficiency assessment of laboratories according to each criterion applied in the proficiency test.

The proficiency assessment of a laboratory is defined by the comparison of the bias of a laboratory with the established tolerance value. The tolerance value equals to twice the Standard Deviation for Proficiency Assessment (σ_{pt}). A result outside the tolerance interval is considered as untrue and is identified by an underlining.

The z-score corresponds to the proficiency assessment of the laboratory.

Colors are associated with the performance of laboratories:

- result in black => results are in the tolerance interval
- result underlined in orange => untrue result, out of the interval tolerance: positive bias,
- result underlined in pink => untrue result, out of the interval tolerance: negative bias,
- **result in red bold italic** => Incoherent result.

For further information please read page 11 of this booklet.

Statistical parameters

x_{pt} : Assigned value
 $u(x_{pt})$: Standard uncertainty on X
 $s(x_{pt})$: Robust standard deviation
 $p(x_{pt})$: Number of results

σ_{pt} : Standard deviation for proficiency assessment
 VT : Tolerance value
 Max : Maximum value $x_{pt} + VT$
 Min : Minimum value $x_{pt} - VT$
 p_D : Number of results out of the tolerance interval

Laboratories results (confidential code)

x : laboratory results.
 LAB. : Edition code of the laboratory that appears in the report.

CRITERE / CRITERION	DCHLM37*	
Unité / Unit	$\mu\text{g.l}^{-1}$	
VALEUR ASSIGNEE / ASSIGNED VAL		
x_{pt}	8,6	
$u(x_{pt})$	0,5	
$s(x_{pt})$	1,9	
$p(x_{pt})$	26	
APTITUDE / PROFICIENCY		
σ_{pt}	2,2	
VT	4,3	
Max	12,9	
Min	4,3	
p_D	3	
LAB.	x	z
1289	<u>(ED) 13,3</u>	2,19
1294	<u>(MS) 3,4</u>	-2,42
1529	(MS) 9,4	0,37
1543	(ED) 8,9	0,14
1552	(MS) 8,7	0,05
1613	(MS) 9,4	0,37
1691	(MS) 8,9	0,14
1954	(MS) 8,7	0,05

Additional information

For some PT with spiked matrices, the spiking range appears in this table for each parameter.

CRITERE / CRITERION	DCHLM37
Unité / Unit	$\mu\text{g.l}^{-1}$
VD	10,5

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The legend of analytical criteria is described at the end of the results tables. The legend of statistical parameters is detailed in part 4: Annexes.

Laboratories with traceability problem are indicated in the BIPEA comment section; their results are not taken into account for the estimation of the assigned value.

According to the nature of the criterion, the assigned value and the tolerance value will not be estimated in the same way.

The assigned value can be:

- A value from the formulation (by gravimetry for example),
- A consensus value of « expert » laboratories (a group of well-known and controlled laboratories),
- A consensus value from the results of all laboratories.

The standard deviation for proficiency assessment (σ_{pt}) can be:

- A prescription value or a regulatory value,
- A performance aim decided by the participants,
- A value from the reproducibility standard deviation of the standardized analytical method,
- A value from the standard deviation of the results of the participants.

The tolerance value equals to twice the standard deviation for proficiency assessment.

▪ **Determination of a z-score**

When a z-score can be determined, it will appear in the results tables. z-score corresponds to the proficiency assessment of the laboratory. It allows to draft charts showing the ranking of laboratories. (see part Graphical representation of results).

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Laboratories results

CRITERE / CRITERION	DCHLM37*	
Unité / Unit	µg.l ⁻¹	
VALEUR ASSIGNEE / ASSIGNED VAL		
x_{pt}	8,6	
$u(x_{pt})$	0,5	
$s(x_{pt})$	1,9	
$p(x_{pt})$	26	
APTITUDE / PROFICIENCY		
σ_{pt}	2,2	
VT	4,3	
Max	12,9	
Min	4,3	
p_D	3	
LAB.	x	z
1289	(ED) 13,2	2,19
1294	(MS) 3,4	-2,42
1529	(MS) 9,4	0,37
1543	(ED) 8,9	0,14
1552	(MS) 8,7	0,05
1613	(MS) 9,4	0,37
1691	(MS) 8,9	0,14
1954	(MS) 8,7	0,05

z-score for proficiency assessment of the laboratory.

When defined in Commissions, the methods used appear in the results table with abbreviations. See the legend tables in the reports.

Comment:

The z-score is calculated as follows:

$$Z = \frac{x - x_{pt}}{\frac{VT}{2}}$$

Where:

x = Laboratory result

x_{pt} = Assigned value

VT = Tolerance value = 2 x Standard Deviation for Proficiency Assessment. ($\frac{VT}{2} = \sigma_{pt}$).

This z-score is a mode of expression, among others, of a laboratory bias.

In BIPEA PT programs, the calculation of the z-score is based on the assigned value X and the standard deviation for the proficiency assessment.

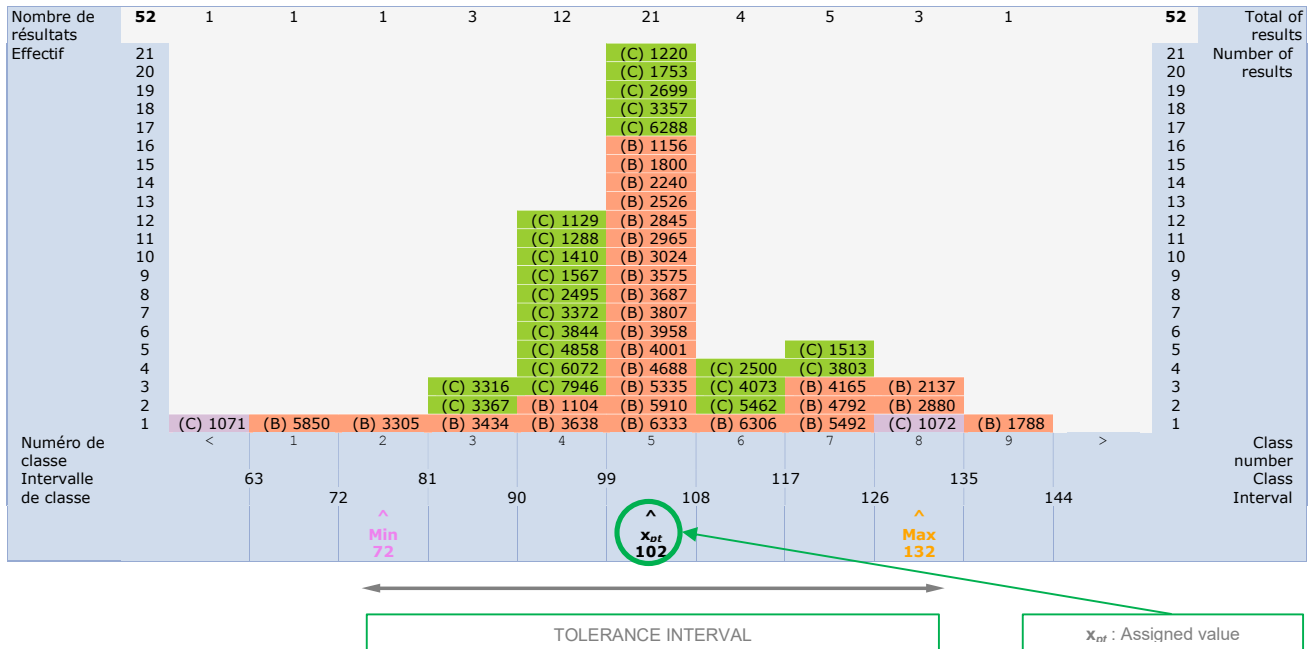
Note: z-score cannot be calculated if $\frac{VT}{2}$ equals to zero, for incoherent results or for not quantitative results.

▪ **Graphic representation of results**

Results are described by graphic form in order to facilitate the reading of laboratories distribution and the methods employed.

Histogram for criterion METH 17 - METHANOL - mg.l-1

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The list of histograms published is determined by the Commission. When different methods or techniques are possible, statistics by method are given as additional information.

There is a description per method with:

- x_m^* = Robust mean
- $u_{x_m^*}$ = Standard uncertainty on the mean
- s_m = Robust standard deviation
- p_m = Number of results

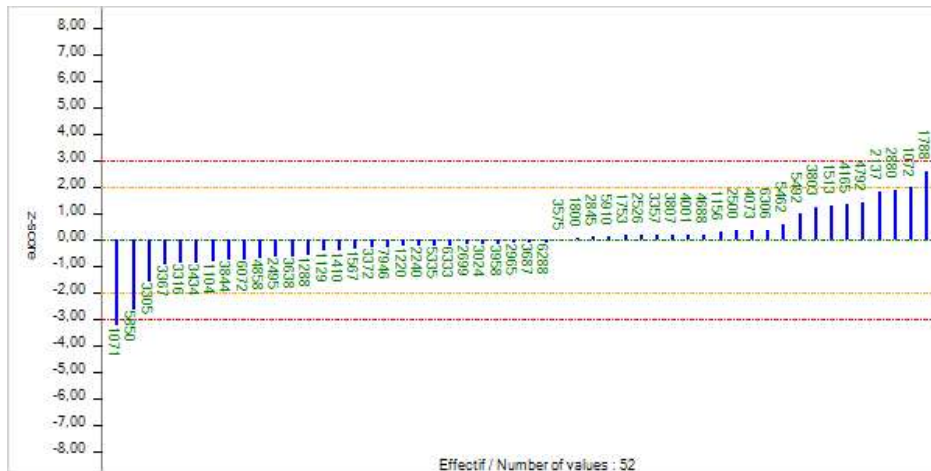
	Méthodes		
	C /Colorimétrie	B /CPG D	C /CPG SD
x_m^*	93	104	99
$u_{x_m^*}$	55	3	2
s_m	63	13	9
p_m	2	28	22

Within the framework of a proficiency testing program, this information does not permit you to compare different methods. However, the differences observed in several tests may provide some valuable trends that can be discussed in Commission.

Graphic of laboratories ranking (z -score)

The report contains a graphical laboratories ranking. This classification includes the z-scores for each laboratory in increasing order.

- Each laboratory can quickly position its z-score in terms of sign and/or absolute value in comparison with:
- z-scores of other laboratories,
- other z-scores on the same criteria, obtained during the previous proficiency tests.



Note: Above 60 results and for an easy reading of the z-score charts, the edition codes of participating laboratories will not appear on the charts.

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ANNEXES

All abbreviations that appear in the report are detailed below.

KEY TO RESULTS TABLES	
CRITERION	List of the analytical parameters proposed in the form. They are identified by a shortened title in the PT scheme report.
ASSIGNED VALUE	
x_{pt}	Assigned value for proficiency testing Value assigned to the analytical parameter for the proficiency test or conventionally true value, usually named "reference value" in the Bipea's PTS. It is most often calculated by the robust algorithm A from ISO 13528 standard.
$u(x_{pt})$	Standard uncertainty of the assigned value It is used to quantify the confidence that can be given to the assigned value. It depends on the mathematical model applied (algorithm A) and is a function of the standard deviation and the number of results used for the estimation of the assigned value. It is calculated as indicated in § 5.6.2 of ISO 13528 standard. Note: If $p_x < 12$, then u_x could not be considered as negligible. This information has to be integrated during the proficiency testing interpretation.
$s(x_{pt})$	Robust standard deviation Standard deviation calculated by the robust algorithm A from ISO 13528 from all the results which participated to the estimation of the assigned value.
$p(x_{pt})$	Number of results for the assigned value Number of results which participated to the estimation of the assigned value.
PROFICIENCY TESTING	
σ_{pt}	Standard deviation for proficiency assessment Characteristic of dispersion related to the evaluation of the results, as defined in ISO 13528.
VT	Tolerance value Two times the standard deviation for proficiency assessment, as defined in ISO 13528. It is a maximum tolerated deviation from the assigned value.
Max	Maximum value $x_{pt} + VT$ Upper limit of the tolerance interval (Assigned value + tolerance value). Value of the analytical parameter over which the result x is considered as untrue.
Min	Minimum value $x_{pt} - VT$ Lower limit of the tolerance interval (Assigned value - tolerance value). Value of the analytical parameter below which the result x is considered as untrue.
p_0	Number of untrue results Number of results out of the tolerance interval.
RESULTS	
LAB.	Laboratories List of laboratories registered for the test. They are identified by their edition code, which is the one of the current series and is available on Bipea's website, button "member area".
x	Result Measurement result.
z	z-score $z = \frac{x - x_{pt}}{\frac{VT}{2}}$ Expression of the result of the laboratory as a value without unit, calculated using the assigned value and half of the tolerance value.
13.57	Incoherent result Result not considered for the estimations and on which no proficiency assessment has been performed. The result is removed through tests of coherence.
13.57	Untrue result Result out of the tolerance interval by overestimation. It corresponds to a positive difference with the assigned value.
13.57	Untrue result Result out of the tolerance interval by underestimation. It corresponds to a negative difference with the assigned value.
< 13.57	Limit of quantification Value of the limit of quantification of the laboratory. This value can neither be taken into account for the estimation of the means and standard deviations nor compared to the tolerance interval.
LD LQ	(L ₀ L _{0Q}) Limit of detection and quantification. Note: For further information, please see Bipea Contact letters No.94, No.98 and No.100.

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ESTIMATION OF ASSIGNED AND TOLERANCE VALUES

You will find at the end of the report, the type of estimation of assigned and tolerance values applied according to the Commission decisions (see table below).

Critères / Criteria	Code / Code	Unité / Unity	Mode d'estimation de la valeur assignée / Assigned value estimation method	Mode de calcul de la valeur tolérance / Tolerance value calculation method
MASSE à l'HECTOLITRE MASS PER HECTOLITER	HECT01	kg.hl ⁻¹	Méthode de référence <i>Reference method</i>	0.8 en valeur absolue <i>0.8 in absolute value</i>
TENEUR en EAU MOISTURE CONTENT	PSECH01		Pas de valeur assignée <i>No assigned value</i>	Pas de tolérance <i>No tolerance</i>
TENEUR en EAU MOISTURE CONTENT	EAUB01	%	Groupe témoin <i>Reference labs</i>	0.30 en valeur absolue <i>0.30 in absolute value</i>
TENEUR en EAU MOISTURE CONTENT	EAUA01	%	Méthode de référence - EAUB01 TENEUR en EAU <i>Reference method - EAUB01 MOISTURE CONTENT</i>	0.30 en valeur absolue <i>0.30 in absolute value</i>
TENEUR en PROTEINES PROTEIN CONTENT	PROT01	%	Population totale <i>All laboratories</i>	2.8 % de la valeur assignée <i>2.8 % of the assigned value</i>
TENEUR en PROTEINES PROTEIN CONTENT	PROA01	%	Méthode de référence - PROT01 TENEUR en PROTEINES <i>Reference method - PROT01 PROTEIN CONTENT</i>	2.8 % de la valeur assignée <i>2.8 % of the assigned value</i>
TENEUR en CENDRES ASH CONTENT	CEND01	%	Population totale <i>All laboratories</i>	0.06 en valeur absolue <i>0.06 in absolute value</i>
TENEUR en CENDRES ASH CONTENT	CENDA01	%	Méthode de référence - CEND01 TENEUR en CENDRES <i>Reference method - CEND01 ASH CONTENT</i>	0.06 en valeur absolue <i>0.06 in absolute value</i>
TENEUR en CENDRES ASH CONTENT	CNDTMP	°C	Pas de valeur assignée <i>No assigned value</i>	Pas de tolérance <i>No tolerance</i>
INDICE de CHUTE selon HAGBERG-PERTEN HAGBERG-PERTEN FALLING NUMBER	EAUBT01	%	Pas de valeur assignée <i>No assigned value</i>	Pas de tolérance <i>No tolerance</i>
INDICE de CHUTE selon HAGBERG-PERTEN HAGBERG-PERTEN FALLING NUMBER	MESS01	g	Pas de valeur assignée <i>No assigned value</i>	Pas de tolérance <i>No tolerance</i>
INDICE de CHUTE selon HAGBERG-PERTEN HAGBERG-PERTEN FALLING NUMBER	TYP01		Pas de valeur assignée <i>No assigned value</i>	Pas de tolérance <i>No tolerance</i>
INDICE de CHUTE selon HAGBERG-PERTEN HAGBERG-PERTEN FALLING NUMBER	HAGB01	s	Population totale <i>All laboratories</i>	18 % de la valeur assignée ; Valeur de tolérance minimum = 14 ; Valeur de tolérance maximum = 72 <i>18 % of the assigned value ; Minimum tolerance value = 14 ; Maximum tolerance value = 72</i>
INDICE de CHUTE selon HAGBERG-PERTEN HAGBERG-PERTEN FALLING NUMBER	HAGBA01	s	Méthode de référence - HAGB01 INDICE de CHUTE selon HAGBERG-PERTEN <i>Reference method - HAGB01 HAGBERG-PERTEN FALLING NUMBER</i>	18 % de la valeur assignée ; Valeur de tolérance minimum = 14 ; Valeur de tolérance maximum = 72 <i>18 % of the assigned value ; Minimum tolerance value = 14 ; Maximum tolerance value = 72</i>
INDICE de SEDIMENTATION SEDIMENTATION VALUE	ZELE01	ml	Population totale <i>All laboratories</i>	10 % de la valeur assignée ; Valeur de tolérance minimum = 3 <i>10 % of the assigned value ; Minimum tolerance value = 3</i>

All laboratories: Value estimated from the results of all laboratories.

Reference labs: Value estimated from the results of a group of known and controlled laboratories.

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SPECIFICATIONS OF SOME PT PROGRAMS

For some proficiency testing programs, other information may appear in the report:

Environment PTS - Characterization of matrices before spiking:

This information is given in a table located in the chapter « 2/ PROFICIENCY TEST ITEMS » in the part « product » of the report.

2/ PROFICIENCY TEST ITEMS

Product

The product analyzed in October 2017 is: Spiked surface water.

Stabilization with sulfuric acid pH <2.

The unspiked surface water matrix used to manufacture the samples for the test of October 2017 of the PTS 37 – Fresh waters – Micropollutants has been characterized by an accredited laboratory.

The results on this matrix are as follows:

Criterion	Unit	Method	Results
pH measurement temperature	°C	In-house method	20.1
pH	pH unit	NF EN ISO 10523	8.3
Conductivity at 25°C	µS/cm	NF EN 27888	710
Suspended solids	mg/L	NF EN 872	8
Nitrates	mg NO3/L	NF EN ISO 10304-1	13
Chlorides	mg Cl/L	NF EN ISO 10304-1	24
Sulphates	mg SO4/L	NF EN ISO 10304-1	73
Calcium	mg Ca/L	NF EN ISO 11885	110
Magnesium	mg Mg/L	NF EN ISO 11885	22
Sodium	mg/L	NF EN ISO 11885	12
Haze	FNU	NF EN ISO 7027-1	3.7
Total Organic Carbon	mg/L	NF EN 1484	3.1
Hydrogen carbonates	mg/L	NF EN ISO 9963-1	354

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