



GENERAL INFORMATION

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INTRODUCTION

Quality Assurance has become crucial for any trade. **Analytical Laboratories**, especially in the **field of Environment**, are subject to this evolution. Whether they are public or private, service provider or industrial, laboratories are concerned with **Accreditation** throughout the world: « procedure by which a conformity assessment body formally recognises that an organisation or individual is competent to perform specific tasks »¹ (*ISO /IEC Guide 2*).

ISO/IEC 17025 contains all the requirements that analytical laboratories should meet to prove their competence. The **accuracy**² of produced results should be guaranteed in the first place. Thus, the **validation of methods implemented** and a **permanent quality control** should be undertaken:

- internally, this validation then this control lead to the monitoring of experimental error (random error);
- externally, they lead to the monitoring of systematic error (bias).

Created in 1993 to overcome the absence of professional provider of External Quality Control in France, the **Association A.G.L.A.E.** has quickly become the first national network of proficiency testing schemes working continuously in the field of the Analysis of Environment.

¹ In France, the only body signatory of the multilateral agreement "European cooperation for Accreditation" is the COFRAC; the COFRAC is the only national accreditation body recognised by the French Government.

² As defined in ISO 5725-1: 1994.

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1°WHAT IS A.G.L.A.E.?

1.1 STRUCTURE

A.G.L.A.E. stands for **Association Générale des Laboratoires d'Analyse de l'Environnement** (General Association of Analytical Laboratories of the Environment). This is a **non-profit-association**, governed by the French law of 1 July 1901 and founded on 16 December 1993.

The Association comprises a General Assembly (all the members), an Administration Board, an Executive Committee and a Technical Committee, as well as the salaried employees (permanent staff). Permanent staff is directly employed by the Association.

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1.2 ACTIVITY

The main activity of the Association is to provide External Quality Control. Proficiency testing schemes are aimed at assisting laboratories to improve their Quality Assurance system and at warning responsible staff against any possible drift of their analytical system.

A.G.L.A.E.'s activity has been expanding since 1993 and covers today various sectors:

- ▲ **physical chemistry** in natural waters (waters intended for human consumption and surface waters), waste waters and saline waters,
- ▲ **measurement of global indicators** in surface waters and waste waters,
- ▲ **measurement of mineral and organic micropollutants** in natural waters and waste waters,
- ▲ **measurement of disinfection by-products** in clean waters,
- ▲ **measurement of pharmaceuticals** in natural waters,
- ▲ **measurement of dioxins, furans and PCB in Biota**,
- ▲ **physical chemistry**, as well as **mineral and organic micropollutants** in various solid matrices (sediments, sewage sludges from water treatment plants, wastes and soils),
- ▲ **ecotoxicology** in surface waters and waste waters,
- ▲ **microbiology** in natural waters, bathing waters and waste waters,
- ▲ **microbiology in sewage sludges** from water treatment plants,
- ▲ enumeration of **pathogenic micro-organisms** in natural waters, bathing waters and waste waters,
- ▲ enumeration of **parasites** in surface waters,
- ▲ **measurement of cyanobacterial toxins** in natural waters,
- ▲ **hydrobiology**,
- ▲ **ecotoxicity and biodegradability of chemical organic compounds in the frame of the « REACH » regulation.**

Key figures: A.G.L.A.E. represents today **500 members, 550 sites including 10%** of laboratories worldwide for a total of **8 000** parcels sent every year. Our scheme includes about **110 proficiency tests**, with **1 200 parameters** statistically processed.

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A.G.L.A.E. also organises **collaborative studies**. These studies are commissioned by third-parties (*French standards body, Ministries, Water Agencies, etc.*) and the objectives may be:

- ▲ characterisation of analytical methods (in terms of linearity, specificity, robustness, etc.);
- ▲ validation of methods (considers more specifically the determination of accuracy) in order, for example to standardise it;
- ▲ validation of alternative methods in relation to reference methods (generally standardised).

A.G.L.A.E. also provides for its members **additional test materials**. These test materials have been prepared in excess so that they may be distributed after the proficiency test to enable laboratories to improve.

1.3 QUALITY POLICY

A.G.L.A.E. Association has been involved in the implementation of a quality system since several years in order to improve the quality of its services. The quality assurance system was first certified according to ISO 9002 standard for three years, then has acquired two accreditations provided by two accreditation bodies BELAC and COFRAC.

Since 1 November 2005, **the Association is accredited by COFRAC** in compliance with ISO/IEC 17025 in combination with ISO/IEC Guide 43-1 and following recommendations of ILAC G13 for the provision of interlaboratory comparisons under the accreditation number 1-1664.

The provisions of these international documents are translated by COFRAC in the document LAB CIL ref 02.

(Accreditation No. 1-1664 – scope available on www.cofrac.fr)



Accreditation No. 1-1664

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2° ORGANISATION OF THE SCHEME

2.1 IMPLEMENTATION OF THE SCHEME

A.G.L.A.E. implements every year Proficiency Testing Schemes for its members.

The content of the scheme is set by the members of the Association; within the ordinary General Assembly. The members of the Technical Commission and the permanent staff of the Association, in line with the Administration Board, come up with a first suggestion. This suggestion is used as a basis of discussion during the plenary meeting.

When the members have expressed their wishes, the Association studies the suitability then implements the final scheme with the selected subcontractors.

Indeed, the Association does not prepare the test materials. Specialised subcontractors are selected, based on their competence and renown.

Once the modalities of the scheme are set, proficiency tests are implemented according to a specified schedule from **February to January**.

2.2 IMPLEMENTATION OF PROFICIENCY TESTS

The main steps of our proficiency tests are the following:

2.2.1 PREPARATION OF TEST MATERIALS

Test materials are prepared and packaged by A.G.L.A.E.'s subcontractors following our prescribed requirements (concentration range, flask and packaging...). These subcontractors are called **Operators**.

Test materials are **close to the ones routinely analysed** and mainly prepared from natural matrices. An adjustment (spiking and / or dilution) can be realised to bring the concentration level of the parameters to the wished values. Samples sent to participating laboratories are mainly **test materials ready to use**.

2.2.2 TRANSPORT OF TEST MATERIALS

Test materials are then transported to participating laboratories by a courier selected for its reliability. The service offer selected for France (Metropolitan France and Corsica) is a delivery the day following the sending. For laboratories outside France, a customised contract review is necessary.

☞ Thank you to contact us for the delivery time.

2.2.3 RECEIPT AND ANALYSIS OF TEST MATERIALS

Conditions of storage and analysis of test materials, as well as the deadline to carry out the analysis, are given in an instructions' form sent prior to distribution.

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Note that a **reasonable period to start the sample treatment** is recommended for each parameter. This period ensures the stability of test materials. Therefore analyses should be carried out during these periods given in the document Er-5-06-EN "Technical Description".

2.2.4 CONFIDENTIALITY OF TEST RESULTS

The Association implements the necessary procedures to protect its members from any misuse of the data concerning them. Results belong to the concerned laboratories only.

Anonymity and confidentiality are secured. Anonymity by not disclosing the participants' list, and confidentiality by **hiding participants' identity**. For this matter, an automatic application replaces the name of the laboratory by a unique and personal laboratory code.

During a proficiency test, you will just have to return your results by email at a dedicated email address. The automatic application will code without human action your results and inform you about your code for this proficiency test.

To summarize, no relation is possible between the name of a laboratory and its results.

2.3 RETURN OF INFORMATION

2.3.1 REVIEW OF TEST RESULTS

The week following the closing of a proficiency test, the Association sends to participants a **review of the test results**. This first feedback enables laboratories to respond promptly to any major anomaly in the quality of their analyses. For proficiency tests involving chemical parameters, a preliminary z-score as well as a first estimate of the assigned value and of the results' dispersion are provided.

2.3.2 STATISTICAL PROCESSING

Statistical data processing is realised with results obtained from **all of the methods combined** and in accordance with precise rules written in standards or in reference documents.

Performance statistics are determined by **consensus of the results of participants who analysed during the reasonable period to start the sample treatment**; these statistics include the z-score mainly but also the mean and the standard deviation of the data dispersion of the proficiency test.

Trueness is approached only when spiking has been used.

Analytical performances of laboratories are expressed with a **z-score** reflecting the deviation incurred in the results from the assigned value (laboratory bias). The main quality of this indicator is its direct and invariant interpretation.

To this z-score is added a qualitative **ranking** that expresses the analytical performances of laboratories as a « grade », taking into account other factors, such as the repeatability of the laboratories' results or the **quality of test materials provided** (refer to the annexes). *[A guide to interpret the test reports (Er-6-29) is available to our members on the web site].*

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2.3.3 TEST REPORTS

About **7 weeks after the reporting deadline**, a **complete test report**, at the pdf format, is issued.

Test reports present three main parts:

- first part: **presentation and interpretation of the proficiency test.**
The conduct of the proficiency test is summarized. Who prepared the samples? How? What was their quality (stability and homogeneity)? Any interesting observations (differences between methods for example)?
- a second part including:
 - ✓ **tables presenting the synthesis of the data processing results.** These documents aim to give an overall picture of the proficiency test.
 - ✓ **tables and charts giving the details of the data processing results** (refer to the examples in annexe).
- a third part with a **synthesis report**, which is an overview of the main information of the test report.

2.3.4 THE EVALUATION OF UNCERTAINTY OF MEASUREMENT

ISO/IEC 17025, which is used for the accreditation of analytical laboratories, led chemists to consider the uncertainty of measurement for all the measurements they produce. Accredited laboratories must indeed be capable of giving their uncertainty of measurement, for instance during audits.

More generally, **the control of the accuracy of measurements has become a necessity.**

Ordering parties (local authorities, water agencies, ...) and industrials are more and more requiring information related to the « imprecision » of the measurement results that are sold to them. This is quite understandable: how to deal objectively with risk assessment without knowing the variability of the criteria on which the decision is taken?

Proficiency testing schemes are one tool to estimate the uncertainty of measurement. French standard XP T 90-220 (August 2003) « Water quality – Protocol to estimate the uncertainty of measurement associated with an analysis result for physicochemical analytical methods » includes a specific chapter dedicated to **proficiency tests as a way to estimate its own uncertainty of measurement** (§ 5.5 of this standard). For this matter, A.G.L.A.E. provides tools (Excel files available on our web site) for its members so that they can use their z-scores to calculate their own uncertainty of measurement.

In microbiology, A.G.L.A.E. takes an active part in standardization works on the uncertainty of measurement, at the national and international level. The implementation of specific training sessions on this subject has already enabled a large number of laboratories to cope with this issue.

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2.3.5 ANALYSIS OF TECHNICAL INFORMATION

Statistical processing of our proficiency tests is not limited to the calculation of precision values and to the evaluation of participants' analytical performances. A.G.L.A.E. also reports « technical » observations, such as:

- instability of barium in the presence of sulphates even in samples stabilised by concentrated HNO₃ (also for silver in the presence of Cl⁻);
- differences between **analytical methods**, for example **EN ISO 11885 of 1998 (ICP-OES) and EN ISO 15586 of 2004 (AA-furnace)** for the analysis of metals;
- differences between extraction methods on solid matrices;
- highlight of differences between microtiter plates types for MPN technique;
- observations of differences between culture media suppliers for microbiological enumerations.

**For more information:
contact us**

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ANNEXE 1: EXAMPLE OF TABLE OF RESULTS AND PERFORMANCES*

Laboratory code	Flask A		Flask B		Observed precision		z-score	Qualitative Ranking
	Replicate 1	Replicate 2	Replicate 1	Replicate 2	Sr	Mean		
1	108,0	99,0	106,0	103,0	4,74	104,00	+0,14	A
2	92,0	91,0	91,0	89,0	1,12	90,75	-0,92	A
3	97,0	94,0	94,0	96,0	1,80	95,25	-0,56	A
4	104,9	101,2	92,2	96,5	2,84	98,70	-0,28	A
5	100,4	100,8	104,8	104,0	0,45	102,50	+0,02	A*
6	95,0	94,0	94,0	97,0	1,58	95,00	-0,58	A
7	96,2	95,5	94,9	94,2	0,49	95,20	-0,56	A*
8	108,1	107,9	109,4	106,0	1,70	107,85	+0,45	A
9	88,2	87,7	111,1	117,9	3,41	101,23	-0,08	A
10	78,7	79,0	77,5	78,7	0,62	78,48	-1,91	A*
11	107,0	104,0	145,2	155,0	5,12	127,80	+2,05	B
12	100,3	100,2	100,1	99,6	0,25	100,05	-0,18	A*
13	100,4	98,4	99,2	94,9	2,38	98,21	-0,32	A
14	150,0	160,0	165,0	140,0	13,46	153,75	+4,14	C
15	87,0	89,0	89,4	93,4	2,23	89,66	-1,01	A
16	124,1	126,8	100,3	99,2	1,46	112,60	+0,83	A
17	68,1	71,5	70,5	70,7	1,70	70,20	-2,57	B
18	107,5	111,5	108,0	108,0	2,00	108,75	+0,52	A
19	104,0	113,2	116,8	114,4	4,75	112,10	+0,79	A
20	115,4	115,2	116,6	117,2	0,32	116,10	+1,11	A*
21	74,6	89,8	74,1	89,9	10,96	82,10	-1,62	C
22	98,0	105,0	95,0	101,0	4,61	99,75	-0,20	A
23	181,5	183,5	185,3	186,0	1,06	184,08	+6,58	C
24	95,0	93,0	112,0	111,0	1,12	102,75	+0,04	A
25	132,2	136,4	133,5	136,4	2,55	134,63	+2,60	B

Metals in clean waters

Parameter	Al
Unit	µg.L ⁻¹

☞ Please refer to the following page for the explanations.

* Please note that this table is a limited example. The number of participants in these proficiency tests « metals in clean waters » is in fact **one hundred** or so.

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Interpretation of the table « Results and performances »

☞ Precision observed:

- **Sr (laboratory standard deviation of repeatability)**: standard deviation of the results obtained by a laboratory under repeatability conditions.
- **Mean**: this is the mean of the obtained results by a laboratory, expressed in the unit of the parameter.

☞ **z-score** is the result position of each laboratory compared to the overall mean of the proficiency test (laboratory bias). The result is all the more satisfactory that it is close to 0. Conversely, the result is all the more unsatisfactory that it is far from 0.

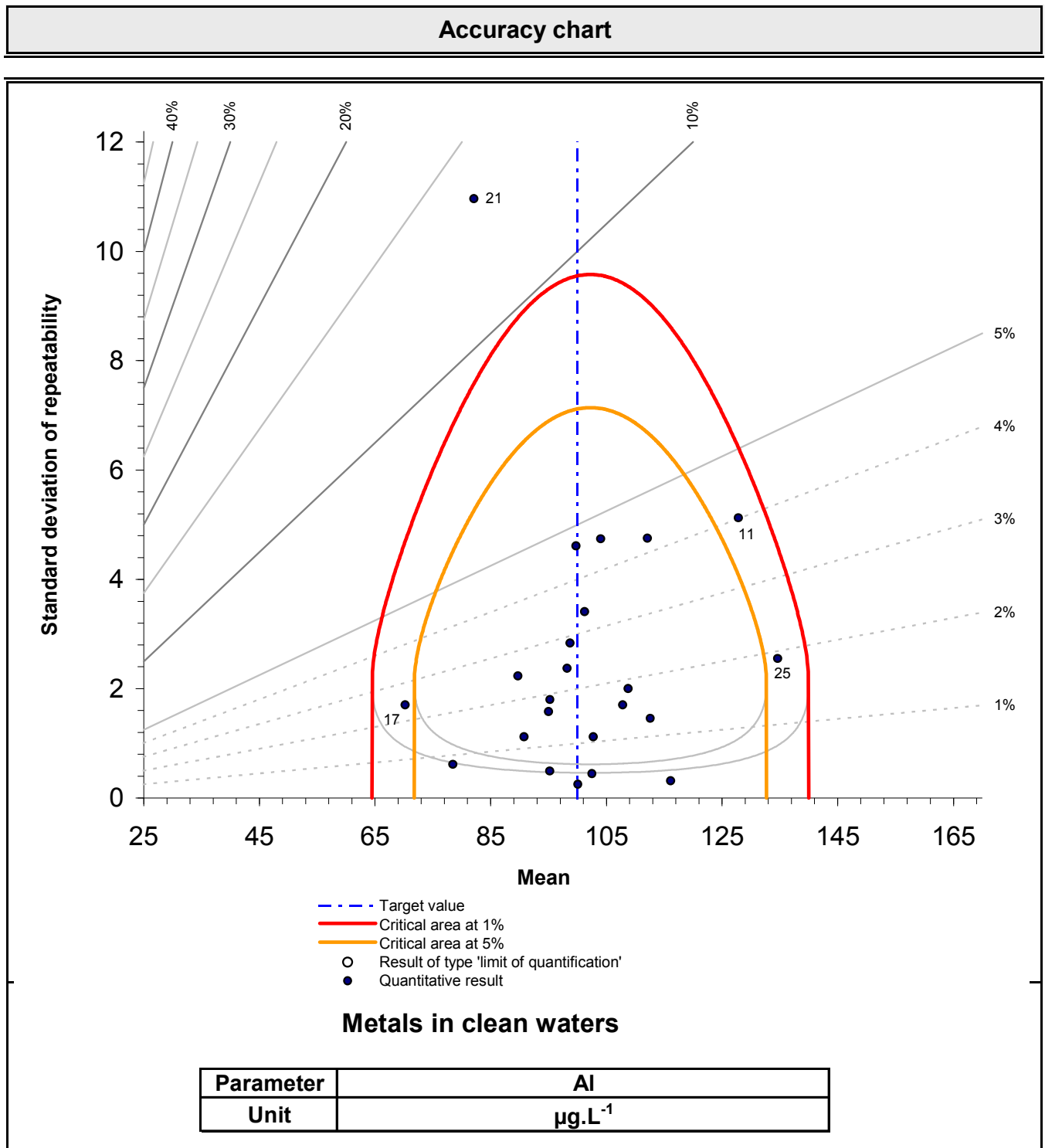
☞ **Qualitative ranking** expresses as a « grade » the quality of the analysis performed by each participant, for each parameter. This « grade » is expressed with a « Letter » code and a « Colour » code.

Qualitative ranking positions laboratories relative to the accuracy of their analysis, that is trueness and precision.

Frequent qualitative rankings are the following ones:

Code	Interpretation
A	Accuracy of the result satisfactory
B	Accuracy of the result questionable
C	Accuracy of the result unsatisfactory
A*	Accuracy of the result satisfactory with a low repeatability
B*	Accuracy of the result questionable with a low repeatability
LQ(A)	Accuracy of the result of type « Limit of quantification » satisfactory
LQ(B)	Accuracy of the result of type « Limit of quantification » <i>a priori</i> satisfactory
LQ(C)	Accuracy of the result of type « Limit of quantification » <i>a priori</i> satisfactory

ANNEXE 2: EXAMPLE OF ACCURACY CHART (CHEMISTRY)*



* Please note that this chart is a limited example in terms of number of participants.

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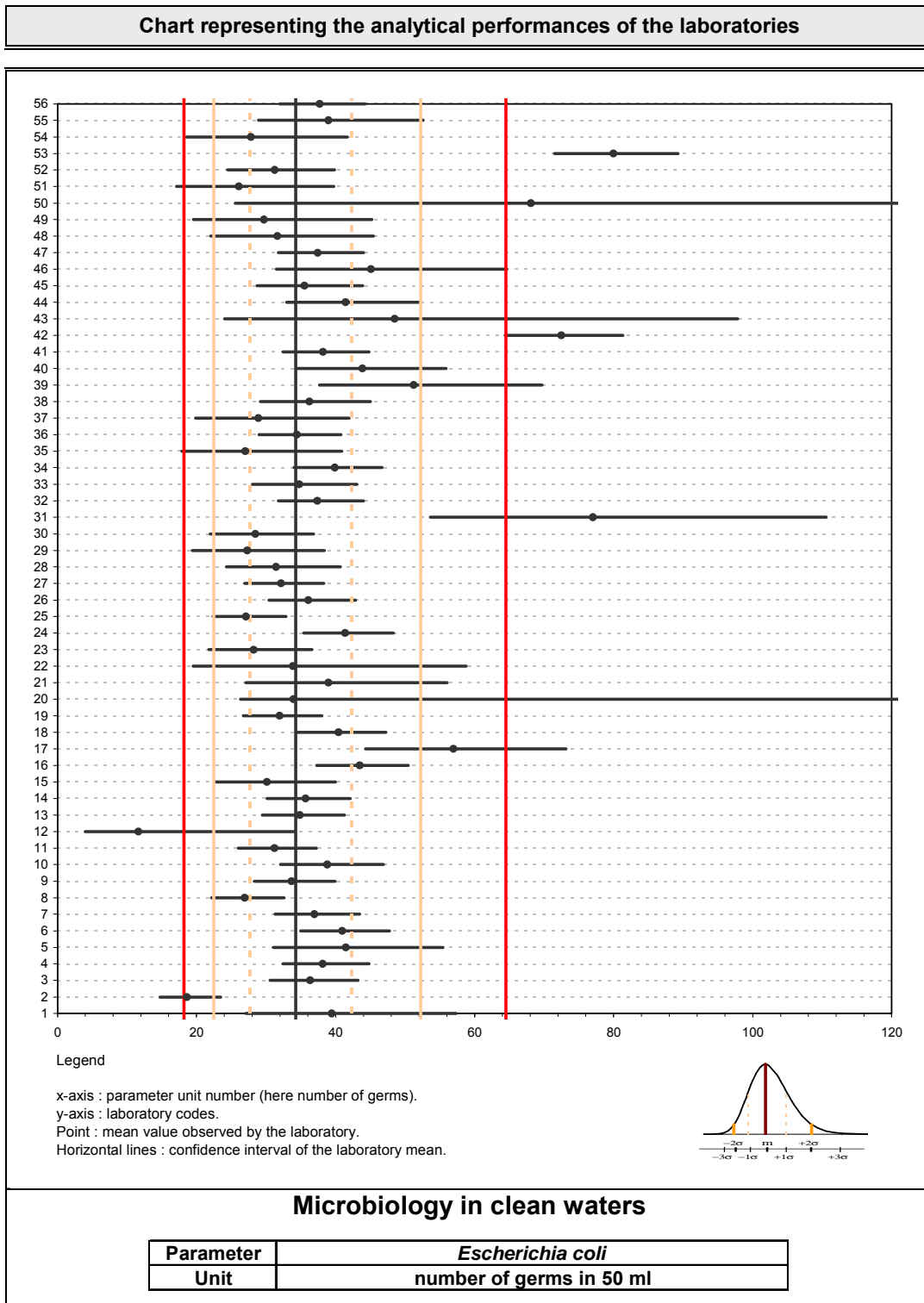
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ANNEXE 3: EXAMPLE OF "CONTROL CHART" (MICROBIOLOGY)*



* Notes of pages 9 and 11 remain valid : in fact, about 190 participants are taking part in these proficiency tests « microbiology in clean waters »

Interpreting the dispersion of laboratories (use of a log-Normal model fitting)

