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INSTITUTE OF APPLIED SCIENCES AND TECHNOLOGY

DEPARTMENT OF PHYSICAL MEASUREMENTS

العربي بن مهيدي أم البواقي .جامعة

معهد العلوم و التقنيات التطبيقية

قســـــم القياسات الفيزيائية

Course of

Metrology 2 quality1

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Semester 3 (2024-2025)

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General introduction

This subject, called metrology 2 quality 1, is intended for second-year students in physical measurements. Indeed, the quality of a measurement in metrology can only be certain if certain conditions and procedures are properly respected. This is the very purpose of this subject that we propose to study in this third trimester. The program is divided into two main parts: metrology and quality. The organization of the booklet is made into seven complementary chapters. In the first chapter, we will see some basic notions on metrology already covered in the first year, the student will have the opportunity to remember how to calculate absolute and relative errors. We will be led to calculate the measurement uncertainty by respecting the calculation guide recommended by the OECD (GUM method based on known statistical methods such as that based on the calculation of the standard deviation). In chapter two, we will discuss F (metrology) in-company and the measurement management system symbolized by the ISO/10012 standard. The third chapter is devoted to calibration and verification documents and their importance in the metrology function. In the fourth chapter, we will discuss the organization of metrology on a national and international level as well as the concepts of accreditation, certification, connection and traceability. The previous chapters only concern metrology, those that follow will be devoted to quality assurance. Chapter five introduces the principles of good laboratory practices. The study in chapter six revolves around the introduction of quality standards. Finally, the seventh and final chapter is devoted to the organization of quality in the company through the ISO 9000 standard. The face-to-face course is enriched by series of application exercises and practical work, we have proposed five series of tutorials that can be improved in the future. The tutorials allow students to familiarize themselves with basic calculations according to the old methods and the new methods based on the GUM method. The practical work allows students to process data using the Excel spreadsheet. We have omitted to put the series of directed works and practical works in this booklet to lighten the content, all the series are available online.

A series of practical work is proposed: calculation of the molecular mass of a known chemical compound taking into account the different uncertainties, calculation of the composite uncertainty of different measuring instruments in a chemistry laboratory, calculation of the composite uncertainty during an acid-base assay, calibration of a laboratory measuring device through repeated experiments in order to highlight repeatability. We will end the series of practical work with a concrete example that can concern both the laboratory and industry. Finally, we hope that this document, worked on as briefly as possible, will give students a helping hand in assimilating this subject that is so important in their university curriculum, especially those in professional training.

Lecturer
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1 Introduction

Metrology is a science in its own right, its developments are in continuous progression, and each year new studies will be made bringing improvements and new methods. In this chapter, we will study the new method based on the GUM while reviewing the basic notions already studied in the first semester of physical measurements. This new method is in fact based on the determination of the standard deviation or the variance, two notions already encountered in statistical mathematics.

1.1 Reminders

1.1.1 Expected skills

Errors and associated concepts: "the objective is to find the sources of error which can be several after having carried out a measurement by taking into account the variabilities of the measurement operation and of the phenomenon itself (these are factors which can be linked to the instruments, the operator or the experimenter). These essential basic concepts allow a better vision for any calculation made."

Uncertainties (related concepts): "there are several types of uncertainty; in this case the goal is to compare and evaluate the different values of uncertainty in relation to each source of error that could be observed. It is therefore necessary to know how to evaluate the different sources that relate to it:

It is necessary to know how to evaluate the uncertainty of repeatability while using a previously known evaluation formula, it is necessary to know how to evaluate the initial measurement uncertainty obtained using a well-defined and well-calibrated measuring instrument.

It is necessary to know how to evaluate, using a given formula, the measurement uncertainty calculated after establishing the protocol (in which several types of errors contribute)."

Expression and acceptability of the result: "the goal is to know how to use significant figures and how to write them scientifically, so the following must be respected:

We must imperatively join the calculated uncertainty to this writing,

We must express the/last result obtained from the measurement with a/value coming from an average and a measurement uncertainty in relation with a level of confidence well determined,

Without forgetting the evaluation of the relative precision. Subsequently, we will determine the measurements to keep according to a specific well-defined criterion,

It is important to make some remarks on the result obtained. From any measurement operation by comparing it to a/reference value",

We will conclude by identifying relevant proposals in order to make the procedure even better.

1.1.2 Definitions

The measurement error "is by intuition defined as the difference between the exact value of a quantity and the measured value of this quantity. This measurement error is just an unknown. Finally, to assess the precision of a certain measurement, we must then associate a measurement uncertainty with the measured value."

"...However, the realization of a measurement or simply measurement ***(this definition is applicable to all scientific fields such as physics and chemistry and many others),*** is to seek the value (numerical value) of any quantity; however it is impossible to know if the value is exact (or true value), this is due to measurement or measurement errors. Before any calculation, it is important to determine the causes behind these errors, and then determine the methods in order to evaluate the measurement uncertainties and then, the presentation of the result. Indeed Errors and designations."

1.2 Errors components:

1.2.1 Random errors

"...The random errors are due to the circumstances of the manipulation/to the manipulator himself, the operating conditions ***(pressure and temperature for example)*** which can vary from one experiment to another. It is important to ask certain questions in order to know if we can reduce them ***(these random errors)***. It is necessary to know how to proceed in order to have the smallest possible value because it is impossible to eliminate random errors. Indeed to decrease or reduce random errors, we increase the number of measurements or experiments. So the greater the number of experiments, the smaller the random errors."

1.2.2 Systematic errors

Systematic errors: "errors that are due to the measuring device used in the measuring operation or the measuring process or the experimenter himself. These errors repeat themselves in a recurring manner and at the same time and have constant values. It should be noted that systematic errors could be corrected. Examples of Systematics/errors include the following:

One or more errors in the manufacturing or the design of the measuring device; A false calibration/verification; a fault in the calibration of the measuring device, fault of setting the zero; the conditions of use that may be contrary to the manufacturer's specifications and requirements; Error in reading an indication or even a parallax error."

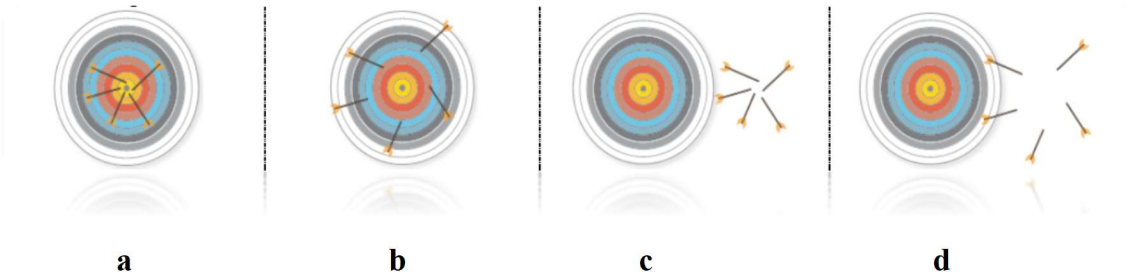
2 Metrology vocabulary

Precision… “The precision can be defined as the quality of a measuring device whose ***systematic errors*** are greatly reduced. We speak about precision when the true value is very close to the measurement act.”

Fidelity… "In this case the representation of the results of ''measurement'' is done as a grouping around their mean value. The ''fidelity'' is then the quality of a measuring device whose ***random errors*** are very low."

Accuracy… "The accuracy is the ability of a measuring device to be both faithful and accurate; it is said to be exact."

- Type of errors and character of the measurements carried out according to the following diagram:



***a*- Measurements accurate and precise** … "Systematic and random errors are low, we can say that the equipment and the operator are good,

***b*- Accurate measurements but not precise**; we can say that we have a good device or equipment but the operator is not very precise or on the contrary we have a good operator but the equipment is not very precise, random error is high in this case comparatively to systematic one…

***c*- Faithful/(precise) measurements**…"but false; we can conclude that systematic errors are higher, we have a good operator but the equipment or the protocol may be defective"

***d*- Wrong / and not very faithful (not precise) measurements**… "We can conclude that systematic errors as well as random errors are very high"

In metrology, it is important to answer the question: ***Is it possible to find or detect systematic error easily?***

"The answer to this question may be: the presence of systematic error is difficult to detect, especially when we do not know where the target is; the dispersion tells us about random errors only."

3 The Uncertainties

3.1 Absolute uncertainty or measurement uncertainty

Any physical quantity having a value "M", this quantity must be accompanied by an absolute uncertainty "Δm"; the latter designates the estimate of the measurement error.

The result will be written as follows: $m = m_o \pm \Delta m$ (g_o is the measured or calculated value). This presentation of the final result means that the true value of m have 95% chance of belonging to $[m_o - \Delta m,\ m_o + \Delta m]$: This interval will be called the confidence interval.

Note:

To a measured quantity M , we add the uncertainty. The latter is noted $U(M)$. This notation means uncertainty in Anglicism.

Uncertainty	Notation	International norm
Standard-uncertainty	s	**u**
Expanded standard-uncertainty	ΔM	**U**
Experimental standard-deviation	s_{exp}	u_{exp} or s_x or σ_{n-1}
Interval width	2 ΔM	q=2a

3.2 Measurement uncertainties and their evaluation

The measurement uncertainties are subdivided into two kind, those that are classified as A and even AS in certain notations and uncertainties type B. We will notice these notations when accessing uncertainties in the chemistry laboratory. On the precision glassware, we can read a lot of information transcribed in blue or brown color. Among this information, we can read the precision next to the volume and the temperature at which this information is valid. We will start with the uncertainties type B then we will see with examples that type/ B.

1. **Uncertainties of type B** :

When statistical determination is impossible, the estimate is said to be class B or /type B. In this case, a single measurement is made where the evaluation of this type of uncertainty must take into consideration the measuring tools (the device) selected by the operator.

In summary, when the measurement is made by a reading on a measuring scale with '95%' for confidence level… then the measurement uncertainty attached to this reading is calculated at the level of half of the smallest graduation.

… When using glassware, its tolerance is considered absolute uncertainty: instruments, not all, made of glass have values printed on the glass by the manufacturer obtained after several tests. Examples of precise glassware include flasks, burettes and pipettes, whether they are gauged or not.

Application example:

We have a graduated pipette, a graduated flask and a graduated burette in a chemistry laboratory. We are trying to specify the maximum volume taken or measured as well as the uncertainty associated with…each/ measuring/ instrument/ used:

- Burette of 50 mL : absolute uncertainty $\Delta V = \pm 0.02$ mL; the volume can be written as $V = 25.00 \pm 0.02$ mL
- Pipette of 25 mL : absolute uncertainty $\Delta V = \pm 0.03$ mL; the volume can be written as $V = 25.00 \pm 0.03$ mL
- Volumetric flask of 25 mL : absolute uncertainty $\Delta V = \pm 0.04$ mL; the volume can be written as $V = 25.00 \pm 0.04$ mL

2. Uncertainty of type A

In the case where "uncertainties" are determined by a 'statistical' method, this evaluation is classified as type uncertainty/A. The latter only relates to measurements that can be made under the same conditions several times. The repetition of measurements several times must be made by the same manipulator *(or experimenter)* under the same experimental conditions. We must follow the same experimental method and the same steps.

The chosen method:

- We carry out a number '**n**' of measurements (limited number) of the same quantity

  $X : x_1, x_2, \ldots, x_n$

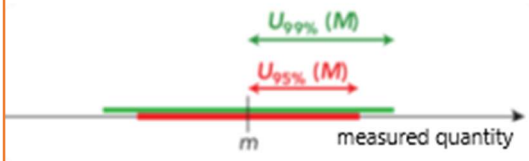
- The best calculation of the "true value" is by using the "average" value of all the measurements which we will note:

$$\overline{x} = \frac{\sum_{i=1}^{n} x_i}{n}$$

- The absolute uncertainty $\Delta \mathbf{X}$ then corresponds to:

$$\Delta x = \frac{1}{n} \times \sigma \ \ with \ \ \sigma = \sqrt{\frac{\sum_{i=1}^{n} (x_i - x)^2}{n-1}}$$

Standard-deviation = repeatability uncertainty to calculate the data dispersion. Absolute uncertainty with confidence level equal to 68 % , the factor =1; to obtain a confidence level of 95%, the factor = 2.



Practice : to calculate the standard-deviation, we can use the calculator turning it to statistical mode or using a sofftware spreadsheet such as Excel or other spreadsheet

Training exercise: A concentration measurement is carried out in the chemistry lab. The values obtained by nine pairs of students are reported in the following table:

Exp n°	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
$c\left(mmol.L^{-1}\right)$	25,51	25,43	25,01	25,06	25,17	25,09	25,17	25,28	25,38

We are asked to calculate:

1. The mean concentration
2. The standard deviation
3. The uncertainty at 95 %:

4 The relative uncertainty

The calculation of absolute uncertainty does not give very precise information on the quality of a measurement. For this, we are forced to define a new variable, which is the relative uncertainty; the latter then allows calculating the precision on the estimated result.

$$\text{Re}\,lative\,uncerta\,\text{int}\,y = \frac{absolute\,uncerta\,\text{int}\,y}{mean\,value}$$

This new variable, which is relative uncertainty, has no units or is dimensionless; it is generally expressed as a percentage. It is noted, according to books and bibliographic references.

"Relative uncertainty (*by analogy with relative error*) is a dimensionless number or without units (*very small and less than 1*) that will be generally expressed as a percentage %."

Note:

- The relative uncertainty may be presented with only one significant digit
- It is smaller when the precision is high

Application exercise: we are asked to estimate the relative uncertainty on the volumes of the application exercise on uncertainties on glassware already seen *(previously)*.

5 Best measure

In this perspective, scientists follow three essential steps:

- looking for sources of uncertainty to keep
- deduction of the final protocol
- assessment of the uncertainty of the result

The first two steps are essential and it is important to carry them out meticulously. Scientists will perform the measurement while noticing what they are doing. As a result, they find the sources of uncertainty to be considered. These uncertainties have an influence on the experimental protocol considered and allow for correction of the latter. Scientists can now think of an experimental protocol in a definitive way. Once the protocol is implemented, a mathematical calculation of the uncertainty follows after choosing the calculation method.

6 Result presentation

6.1 Expression :

$x \pm \Delta x$ or $M = m \pm U(M)$ specifying the unit and the confidence level (at 68% or 95%)

The presentation of the final result is as follows: we keep the last digit which has the same place as the two significant digits of the expression of uncertainty.

Application example :

Y = 315,458 U = 2,4 we keep : Y = 315,5 +/- 2,4

T = 1850,6468 K with u(T) = 13,48 K then 14 K (only two significant digits are kept)

Or T = 1851 +/- 14 K (at 95 %)

6.2 Result /Reference:

"The difference between the "***experimental value***" and the "***theoretical value***" divided by the theoretical value expresses a "***percentage %***" and can be given by the following expression":

$$\frac{\left|x_{exp} - x_{\text{theoretical}}\right|}{x_{\text{theoretical}}}$$

Application exercise: we propose to study a series of measurements on the speed of sound, we obtain an average value equal to 347 m.s^{-1}, while the reference value is equal to 340 m.s^{-1}.

$$\frac{\left|v_{exp} - v_{theoretical}\right|}{v_{theoretical}} = \frac{\left|347 - 340\right|}{340} = 2.10^{-2} = 2\%$$

6.3 Improvement proposals:

Among the proposals to improve the calculations:

1 Improper use of the equipment chosen or used incorrectly may be due to a calibration or calibration problem,
2 Experimental method to be revised to improve the final results,
3 In order to minimize random errors, the number of measurements must be increased.

1 Metrology function

In this very important chapter in metrology, we will see some notions related to the metrological function in companies. Among these points, there are the "*concepts*" of guarantee, assurance, traceability, equipment maintenance, measurement, and the necessary "*qualities*" of the personnel responsible for this metrological mission, without forgetting the ISO 10012 standard and the concepts on which it is based.

Introduction

The metrology function is structured around several points to be respected and to answer several very important questions. This goes from the /proper functioning/ of this /metrology function/. The /metrology function/ in a company manages/organizes the equipment fleet and guarantees its compliance with the standards in force. It is to set up a metrology function within any company, this implies first of all giving relevant answers to the following questions:

- What we are looking for in metrological function?
- In addition, what are the objectives?
- Why should we engage in such business?
- What we gain from the metrological function in the company?
- Can the metrological function be subcontracted?

2 Principal missions

The many missions of the metrological function are varied and can be summarized in the following points:

- Control the suitability for use of all measuring equipment present and used in the company…for example the laboratory equipment such as pH-meter, conductivity meter and other apparatus necessary to be used in the laboratory.
- Provide assurance of control of suitability for use (documentary traceability). The documentation is an important parameter in the laboratory or in the company, it provide suitable information about the equipment and the method of use.
- Ensure that this equipment corresponds to its needs (quantity of devices and technical level, etc.). The equipment should be necessary in the laboratory or the company, unnecessary equipment are not needed.
- Guarantee the effective connection of measuring equipment to national or international standards. It is important to connect the measuring equipment to standards (national or international)
- Manage measuring equipment (all actions to be taken to constitute and maintain the fleet of equipment). The management is an important thing in the company; it may ensure an optimal use of the equipment and materials.

3 Fixed missions

Each company has needs in terms of quality and quantity of measuring equipment that must meet precision criteria and comply with standards and norms. This is the function of the metrology mission in the company. Overall, the detailed missions directly related to a metrology department can be summarized in three essential points:

- Tasks related to reference standards
- Tasks relating to measuring devices
- Tasks directly related to company staff (*personnel)*

3.1 Reference standards

A standard has great precision; it is made from precious material (such as platinum) and resistant to various hazards. Its properties allow it to be the object of reference to other instruments, which have less precision being made from less precious materials. Reference standards can be made from solid materials such as weights but can also be in liquid form such as standard solutions in pH-metry.

However, there are precautions to be taken regarding its use and operations involving it. Therefore, the precautions to be taken with reference standards can be listed as follows:

- To receive them (*the reception*)
- To identify them individually (*the registration*)
- To ensure their monitoring (*recording on a life sheet of all operations*)
- To ensure their metrological connection (*the connection*)
- To carry out the necessary calibrations, at the desired frequency (*the calibrations*)
- To ensure that the service provider offers sufficient guarantees of skills (*the guaranties*)
- To ensure their conservation in optimal conditions (*the conservation*)

4 Measuring equipment

A thermometer, for example, is a measuring instrument which gives the temperature of a medium, a flow meter gives the value of the flow in a pipe, a barometer allows pressure to be measured and so for a pycnometer for density measurement.

These instruments and many others make up the measuring means of a company. These then represent the devices that we receive in order to ensure the metrology function. Among the most important points, we can cite:

- Analyzing all the needs related to them and participating in the choice of measurement equipment or apparatus
- Receiving and put them into service (*the reception operation*)
- Identifying (*Matriculation operation*) them individually (*registration*)
- Ensuring their monitoring (*recording on a life sheet of all operations*)
- Determining the type of metrological monitoring to be considered
- Determining the verification and/or calibration frequencies (*it's important to note the dates and the time of calibration/verification in a specific manual*)
- Ensuring their calibration, their verification (*proof of metrological connection*)
- Trigger the necessary repairs

- Determining the measurement uncertainties (*class of devices, A or B for example*)
- Managing, in agreement with users, the purchase of new equipment
- Ensuring the disposal of end-of-life materials

5 The staff (the personnel)

It is important to consider the personnel involved in the metrological function by ensuring their training and monitoring to improve this metrology function in the company...for this it is necessary to ensure:

- Check training and knowledge continuously to update the considered staff of the company (*Even by mean of internships programs*)
- Monitor training and qualifications (*training table*)
- Provide training for metrology staff, as well as for staff from other interested departments

6 Metrologist qualities

"The metrologist is dedicated to metrology and has the role of taking measurements, calibrating and optimizing devices. He calibrates the devices and repairs them; he can operate in the fields of industry (analysis laboratories, automobile industry, and pharmaceutical industry, in civil or military aeronautics). He must have qualities that allow him to assume his role in the company, the main qualities of a metrologist in a company are":

- Be organized and rigorous: He will have to set up a documentary structure, a real management of the fleet of measuring devices.
- Be a good communicator: One of the most important tasks, he is in contact with most of the company's departments: management, purchasing, design office, maintenance, quality, control, production, design, logistics, transport...The qualities of a metrologist continued
- Capacity for analysis and adaptation: He must be able to select the information to transmit and adapt his language according to his interlocutors:
- Daily users of measuring instruments (operators, technicians)
- Maintenance technicians
- Manufacturing managers

7 Insurance/Guarantees

In order to make good measurements, it is necessary to guarantee precision and reliability; these qualities of the measurement give assurance to the latter. Indeed, the guarantees that the metrology function provides can be summarized in the following points:

- Reliability of results (publications, testing, etc.)
- Competence and motivation of staff (know-how, internal and external training, etc.)

- Economy (purchases, calibration, time, etc.)
- Competitiveness
- Credibility
- Homogeneity, provision, pooling of equipment
- Responsiveness (faster implementation of a QS, etc.)

8 Norm ISO 10012

The ISO 10012 standard is based on solid concepts in order to allow good quality of actions. Indeed these actions must be effective in order to lead to a good result. These actions can be summarized in the following points:

- This standard concerns all actions that can lead to a result. It is the very motto of this standard that introduces the notion of process.
- This standard concerns the equipment or measuring device but also the method followed as well as the technology used to carry out the measurement.
- This standard has allowed an evolution that places the metrologist at the center of the measurement process, this metrologist must know how to express himself as well on quality as with:
  - The design office for the design,
  - Maintenance for the upkeep and
  - Production for the use of the measuring equipment.
- Finally, the ISO 10012 standard clearly defines all the concepts of measurement uncertainties, customer feedback in order to improve a service or the approval of IT systems.

8.1 Management System

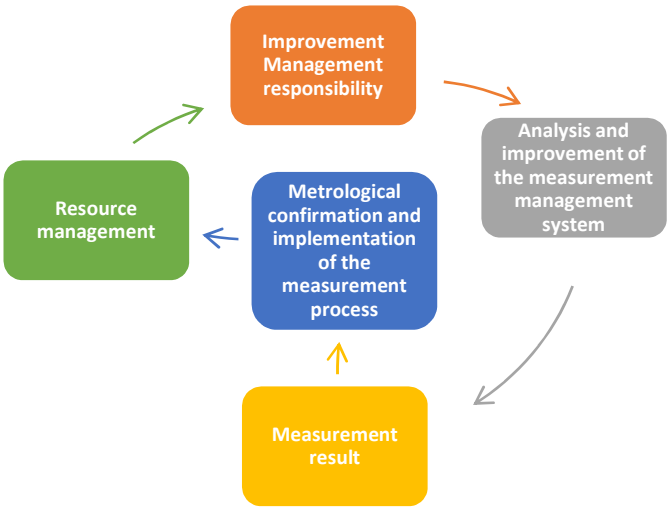


Figure 1 Management system - measurement

1 Documentation

Documents are certificates or records concerning the progress of operations carried out on measuring equipment. This involves identifying the instrument and all its characteristics, its location, its operating instructions and all the instructions relating to it. The technical data sheet and the life sheet are very important in order to know everything about the measuring instrument, from its identification to its tracing.

8.2 Technical sheet

- This is the instrument's identity card; it specifies its location, its technical specifications, the storage and special usage conditions.
- Instructions for use: This document specifies the authorized personnel, the usage standards or test standards as well as the usage precautions (instructions for use, location, configuration, etc.). It indicates the method of use (manual, interfaced, etc.) and the maintenance constraints (cleaning before use, etc.).

8.3 Life sheet

Inside the life sheet we can find the frequency of checks, this check can be internal and/or external, all maintenance actions carried out on the instrument, possible observations and the sheets concerning the anomalies detected if they occur.

Example of life sheet

Life sheet

Material : *Laboratory oven*	Date of commissioning : 0*1/01/2020*
Brand (mark) : *Asus*	Assignment: *laboratory of chemistry*
Type: *T6420*	*Responsible : Laboratory responsible*
Serial number *: 44*	
Inventory number*:* ……	

Verification /calibration /maintenance

Verification/calibration **Internal☐** **External☐**	Reference procedure : *practical guide* Maximum permissible error : ± 5 ° C Field of use : 105 ° C	*Responsible:* *Substitute :*

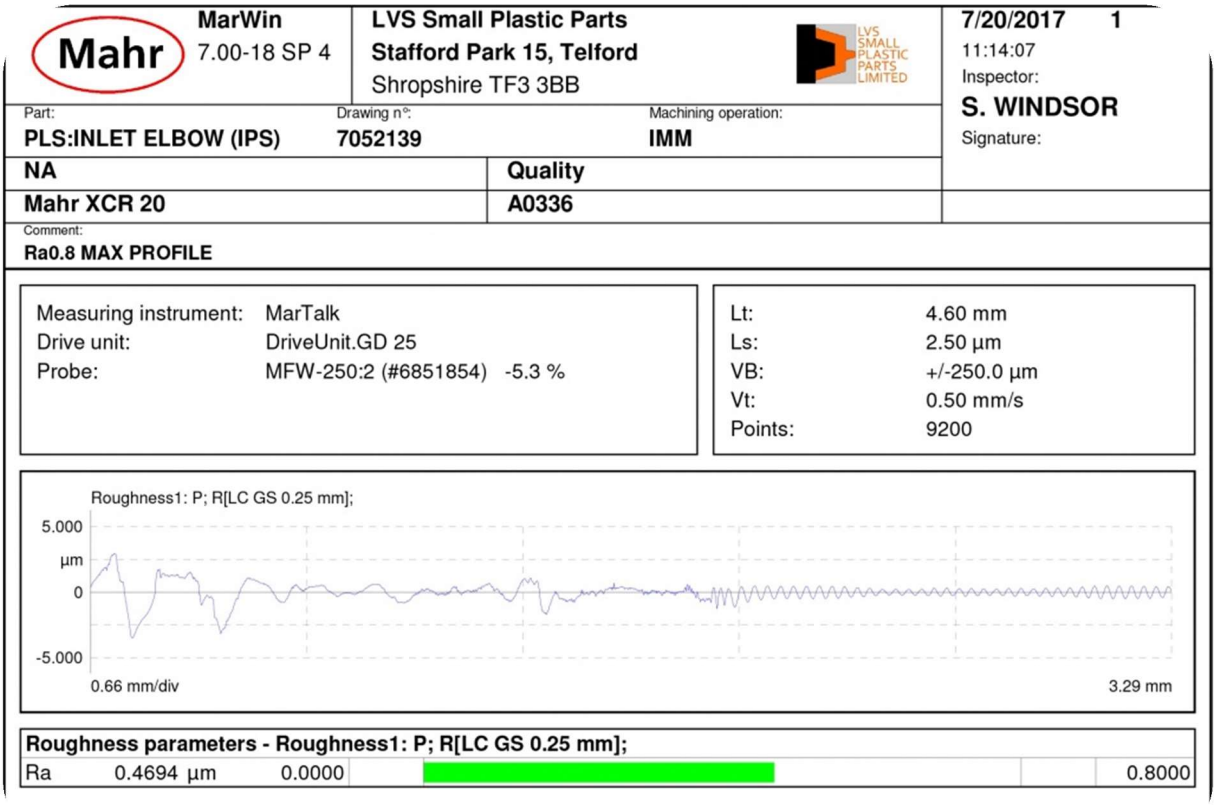
Verification /calibration					**Maintenance**			
Frequency : *1 year*					Frequency : *1 year*			
Date	by	PV N°	Decision	Visa	Date	by	observation	Visa
10/01/2020	…	….	*conform*	….				
22/01/2021	...	….	*conform*	….	25/01/2021	constructor	*RAS*	
23/01/2022	…	….	*conform*	….				

9 Operating mode

The verification procedure includes documents relating to the verification procedure, documents which designate the personnel able to verify a particular instrument, the measurement conditions, the verification frequency, the operations, which must be carried out beforehand, all verification methods, as well as archiving (recording).

9.1 Document-type

It is a document necessary to carry out the verification and can be represented by a spreadsheet in Excel or in another spreadsheet such as Regressi.



1 Metrology organization

Before discussing the organization of metrology and metrological organizations, it is important to discuss measurement unit systems. In fact, we have the following two systems:

- The (SI) system or international system of units
- System of units recommended by the CGPM: General Conference on Weights and Measures.

1.1 Metrological organizations

There are several national and international organizations depending on their mission and areas of expertise. We will limit ourselves to the most important ones:

- *BIPM: International Bureau of Weights and Measures*
- *IEC: International Electrotechnical Commission*
- *ISO: International Standard Association*
- *OIML: International Organization of Legal Metrology*
- *BNM: National Bureau of Metrology*
- *COFRAC: French Accreditation Committee*
- *INM: National Institute of Metrology*
- *LNE: National Testing Laboratory*
- *LCIE: Central Laboratory of Electrical Industries*
- *LPTF: Primary Laboratory of Time and Frequency*

The national and international organizations participating in the calibration chain represent laboratories, which may be at different levels of precision compared to primary laboratories. The latter constitute the pinnacle of measurements (scale of measurements). There are three types of laboratory and each country opts for a two-level system or a three-level calibration system. Many countries (the majority) have chosen the three-level system using three types of singular laboratories:

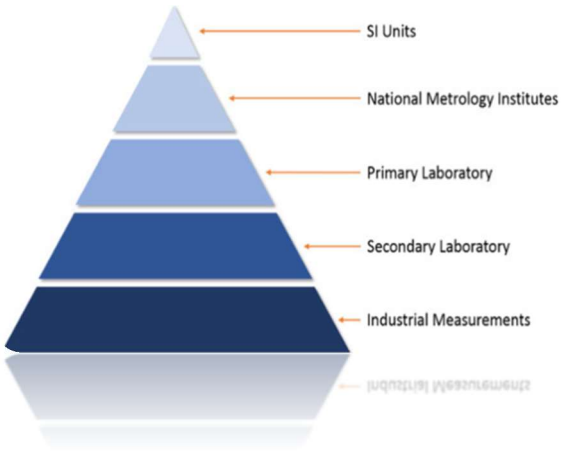
- *Primary laboratory (level 1),*
- *Secondary calibration laboratory (level 2),*
- *Corporate metrology laboratory (level 3)*

1.2 Primary laboratories

"All of these laboratories are composed of reference laboratories (standards) with a national character relating to well-defined quantities."

- Generally, the activity of these laboratories is directly related to research work
- The main subject of the primary laboratories is the carrying out of research work, which makes it possible to reinforce measurement precision and accuracy as well as the conservation of primary standards.

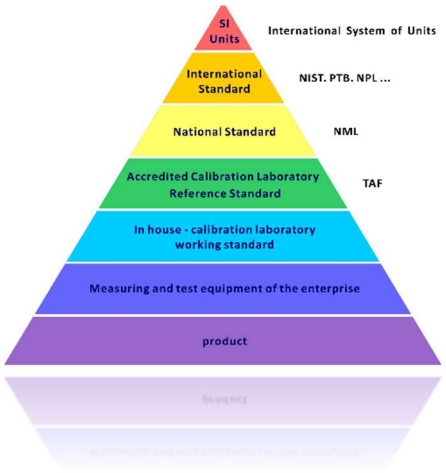
1.3 Secondary laboratories



- They rank second after primary laboratories.
- They define themselves as being organizations responsible for carrying out precision calibrations at the request of laboratories and manufacturers.
- They are the direct contacts of the user or manufacturer of IdMs.
- Their mission is to ensure the connection of any IdM to their reference or secondary standards.
- They are called upon to issue official calibration documents (calibration certificates), after each calibration operation, under the responsibility of the body responsible for supervising the national system of calibration chains.
- To do this, their reference standards must be periodically connected to the reference standards of the primary laboratories.
- These laboratories are used in particular to calibrate the references of the testing and analysis laboratories.

1.4 Tertiary laboratories

- Any company whose metrology plays an important role and which has adequate human and material resources can set up its own laboratory to carry out the calibration of its fleet of instruments and carry out calibrations for third parties.
- The reference standard of this laboratory must be connected to a secondary calibration laboratory, which gives it the tertiary level of precision



1 Good laboratory practices

1.1 The GLP

GLP (or Good Laboratory Practice) encompasses a quality assurance system relating to the management of non-clinical safety studies that have a direct relationship with the environment and health. It is important to highlight the conditions for planning these studies, the conditions for carrying them out, the controls carried out, the methods of archiving or registration, the conditions for recording and dissemination. The studies must be clear and precise.

1.2 Terminology/Test Facility

It (the test facility) is composed of several individuals or persons to which must be added the premises and equipment necessary for carrying out the pre-clinical safety study. The pre-clinical study is directly related to the environment and health. For carrying out studies on several sites, the test facility is composed of the site where there are:

- *The test facility includes the people, premises and equipment that are necessary for the conduct of the non-clinical safety study relating to health and the environment. For multi-site studies, conducted at more than one site, the test facility includes the site where the Study Director is located and all other test sites, which may be considered individually or collectively as test facilities.*
- *The test site includes the location(s) at which one or more phases of a given study are conducted.*
- *The management of the test facility includes the person or persons with formal authority and responsibility for the organization and operation of the test facility, in accordance with these Principles of Good Laboratory Practice.*
- *The management of the test site includes the person or persons (if designated) responsible for ensuring that the phase or phases of the study for which they are responsible are conducted in accordance with these Principles of Good Laboratory Practice.*
- *The ordering party is the legal entity that commissions, sponsors or submits a non-clinical safety study relating to health and the environment.*
- *The Study Director is the person responsible for the overall conduct of the non-clinical safety study relating to health and the environment.*
- *The Principal Investigator is the person who, in the case of a multi-site study, exercises, on behalf of the Study Director, well-defined responsibilities for the phases of the study delegated to him. The Study Director may not delegate to the Principal Investigator(s)*

his responsibility for the overall conduct of the study, including approving the study plan, with its amendments, and the final report, and ensuring compliance with all relevant Principles of Good Laboratory Practice.

- *The quality assurance program is a specific system, including the relevant personnel, which is independent of the conduct of the study and is designed to provide the management of the test facility with assurance that these Principles of Good Laboratory Practice are being followed.*
- *Standard operating procedures are documented operating procedures that describe how to perform tests or work details of which are not normally included in the study plan or testing guidelines.*
- *The master plan is a compilation of information intended to assist in the assessment of the workload and the monitoring of studies carried out in a test facility*

1.3 Terminology/non-clinical safety/environment and health

- *A non-clinical safety study related to health and the environment, hereinafter referred to simply as a "study", consists of an experiment or set of experiments in which a test item is examined, in the laboratory or in the environment, with a view to obtaining data on its properties and/or safety for submission to the competent regulatory authorities.*
- *A short-term study is a study of short duration carried out using standard, widely used techniques.*
- *The study plan is a document that defines the objectives of the study and the experimental devices necessary for its conduct, with any amendments that may be made.*
- *An amendment to the study plan is a deliberate change to the study plan after the start date of the study.*
- *A study design deviation is a non-deliberate departure from the study design that occurs after the start date of the study.*
- *Test system means any biological, chemical, or physical system, or any combination of these, that is used in a study.*
- *Raw data means all original test facility records and documents, or certified copies thereof, that result from the original observations and work carried out in a study. Raw data may also include, for example, photographs, microfilm or microfiche copies, data on computer media, observational records on cassette, automatic data recordings, or*
- *any other means of data storage that is known to be capable of ensuring the safe storage of information for a period of time, as described below.*
- *A specimen means any material removed from a test system for examination, analysis, or preservation. The date of commencement of the experiments is the date on which the first data specific to the study are obtained. The date of termination of the experiments is the last date on which data from the study are obtained.*
- *The study start date is the date on which the Study Director signs the study plan.*

- *The study end date is the date on which the Study Director signs the final report.*

1.4 Terminology/Test Element

- *A test item is an article that is the subject of a study.*
- *A reference item ("control item") is any item used to provide a basis for comparison with the test item.*
- *A batch is a specified quantity of a test or reference item that is produced in a well-defined manufacturing cycle so that it is normally uniform in character and should be designated as such.*
- *A vehicle is any agent used as a carrier medium to mix, disperse, or solubilize the test or reference item to facilitate its administration or application to the test system.*

1 Quality standards

The metrological function provides insurance for the management of the risk of having erroneous or incorrect results that can have a negative effect on the quality of the products. The measurement that represents the heart of metrology is intended to help us make the right decisions. Two very important points will be considered in the quality reference:

- *Validation of the conformity of a product*
- *Verification of the capability of a production process.*

"The metrology function is structured around customer satisfaction and feedback and must instantly adapt to the changing and growing demands of customers. It must set up and plan an organization that can answer the following questions":

- Is the required quality well specified by my client?
  - ❖ Very strong disparity depending on the sector of activity. Be careful, this can be dangerous because it generates a subjectivity of decision of conformity, sources of a significant variability (Automotive sector and service…)
- Do I understand my client's requirements?
  - ❖ We must speak a common language. Ex reading of geometric specification (aeronautics/perfumery)
- This problem introduces the question: what means of measurement to retain and who will be responsible for making these measurements (availability, skills)?
  - ❖ The measurement does not bring added value to the product, it is like an insurance, beware of overbidding.

Example: Reading geometric specifications such as in aeronautics or perfumery

– This problem highlights the question: what are the means of measurement to be retained and which person will be responsible for making these measurements in accordance with availability and competence?

Measurement is like insurance but does not bring added value to the product

Conclusion

- It is the implementation of the metrology function that will allow:
  - ❖ *periodic control of measuring equipment*
  - ❖ *having available a real and instantaneous status of the life of the measuring processes used.*
- It is the implementation of paper or digital records that allow traceability of the following areas:
  - ❖ *Electrical diagram/instrumentation of each measuring chain*

- *Technical notice and specification of each component of the measuring chain*
- *List of maintenance service interventions*
- *Configuration sheet for programmable equipment*
- *Training and authorization of personnel to use the measuring equipment.*

The metrology function must therefore have a real QMS specific to its service. This is NF EN ISO 10012; metrology is therefore an important client of the quality service.

1 Organization related to quality

The quality organization is centered on activities that concern quality control, quality management and quality assurance. Any company, small or large, that is interested in a quality approach must do a better job in order to respond to customer feedback.

1.1 Norms ISO 90000 / ISO 90001

- *ISO 9000 norm : encompasses the vocabulary and principles essential to quality management systems*
- *ISO 9001 norm: international standard, it specifies the requirements for quality management systems. It must demonstrate its ability to consistently provide a product that meets customer and applicable regulatory requirements and aims to increase customer satisfaction.*
- *All the requirements of this standard are intended to apply to any organization, regardless of its type, size and the product provided.*

1.2 Process approach

- Process approach: One of the principles established in ISO 9001 is the process approach.

Process: *"set of correlated or interactive activities that transform input elements into output elements"*

- One of the very first requirements is to define all the processes necessary for the quality management system.

1.3 General requirements

Metrology requirements: They are found across several chapters of the standard and may be common to other activities or specific to monitoring and measurement means.

"The organization must:

- *Identify the processes necessary for the quality management system and their application throughout the organization;*
- *Determine the sequence and interaction of these processes;*
- *Determine the criteria and methods necessary to ensure the effectiveness of the operation and control of these processes;*
- *Ensure the availability of resources and information necessary for the operation and monitoring of these processes;*
- *Monitor, measure and analyze these processes;*
- *Implement the actions necessary to obtain the planned results and the continual improvement of these processes."*

"The Organizations must ensure the determination of the criteria and methods essential to ensure high operating efficiency and to guarantee the control of processes. The guarantee of the availability of the means and information essential to the proper functioning of processes and their monitoring is more than necessary. Thus, the monitoring, measurement and analysis of processes are essential. Organizations must imperatively proceed to the identification of the processes necessary for the quality management system and their application throughout the organization. The implementation of essential actions is mandatory in order to obtain pre-

established results and thus continually improve the processes. These organizations are called upon to determine the sequence and interaction of the different processes."

1.4 Process measurement and measurement process

Process measurement: metrology is not only concerned by point e) with the monitoring and measurement of processes, in reality, it is concerned by many more points.

- Measurement process: It is intended to provide support to obtain the quality of the products manufactured by the company. This process consists of many activities :
  • *Expression of need*
  • *Purchase of measuring equipment*
  • *Monitoring of equipment*
  • *Connection*
  • *Marking*
- The customer of this process is, for example, the user of the measurement result.
- Monitoring and measurement devices are mentioned directly in the chapter on "product realization" of ISO9001.
  "The organization must plan and carry out the production and service preparation activities under controlled conditions. These conditions must include, as appropriate:
  - *The availability and use of monitoring and measuring devices;*
  - *The implementation of monitoring and measuring activities*;"

These points are presented as an element of control of the production process and therefore of the conformity of the products. In the ISO 9001 standard, the role of the metrology function in the declaration of conformity of products is very clear.

"The organization must establish processes to ensure that monitoring and measurement activities can be carried out and are carried out consistently with the monitoring and measurement requirements. »

The standard no longer speaks here simply of measuring equipment, but of processes as a whole, which include equipment, but also personnel, methods, etc.

This principle is also applicable to the monitoring and measurement of processes when it is necessary to implement measuring equipment. "In addition, the organization must assess and record the validity of previous measurement results when equipment is found not to conform to the requirements. The organization must take appropriate action on the equipment and on any affected product»

A particularly delicate point to deal with which may lead to:

- Product recall
- Simple information for customers
- Waiver with or without customer notice
- No measurement, because there are no consequences (the deviation on the device having no impact on the validity of the control results) "In addition, the organization must assess and record the validity of previous measurement results when equipment is found not to conform to the requirements. The organization must take appropriate action on the equipment and on any affected product (...)"

The standard therefore requires that the company assess the consequences of such a situation. To do this, it may examine the following points:

- The evolution of the uncertainty of the equipment
- The consequence on the uncertainty of the measurement
- The examination of the possible influence of this new uncertainty on the validity of the controls according to the permitted tolerances"When it is necessary to ensure valid results, measuring equipment must be:
  a) calibrated or verified at specified intervals or before use, against measuring standards linked to international or national measuring standards (where these standards do not exist, the reference used for calibration must be recorded)"
  b) Adjusted or readjusted, as necessary"
  c) Identified in order to be able to determine the validity of the calibration"
  d) Protected against adjustments likely to invalidate the measurement result"
  e) Protected against all damage and deterioration during their handling, maintenance and storage. »

These are the requirements for connection to the calibration and equipment protection chains. "Records of calibration and verification results must be kept."

- This is to ensure documentary traceability. However, these documents are also essential for making corrections, for example (calibration certificate).
- These concepts of control also apply to software, as it is increasingly used in measurement processes. "When used for monitoring and measuring specified requirements, the ability of the software to meet the intended use must be confirmed. This must be done before first use and reconfirmed if necessary."
- As with measuring equipment, software must undergo initial confirmation and, if necessary, periodic confirmation (comparison with other previously validated software, manual verification as necessary, etc.).

1.5 Tricky Points

A particularly delicate point to deal with which may lead to:

- *Product recall*
- *Simple information for customers*
- *Waiver with or without customer notice*
- *No measurement, because there are no consequences (the deviation on the device having no impact on the validity of the control results) "In addition, the organization must assess and record the validity of previous measurement results when equipment is found not to conform to the requirements. The organization must take appropriate action on*
- *As with measuring equipment, software must undergo initial confirmation and, if necessary, periodic confirmation (comparison with other previously validated software, manual verification as necessary, etc.).*

In Conclusion: periodic verification of instruments and their calibrations is done before and during their use. During transport, measuring instruments can be damaged; maintenance

operations must be carried out with great care in order to protect them. If we do not have reference standards, we must proceed with registration. "All calibration verification results must be kept" This concerns the traceability of documents. But these are essential to make the necessary corrections (e.g. the calibration certificate). "The ability of software to meet the requirements of intended use must be confirmed, especially when it is used for the measurement of specified requirements and monitoring. This must be planned before the first use."

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