LARBI BEN M'HIDI UNIVERSITY OUM-EL-BOUAGHI

INSTITUTE OF APPLIED SCIENCES AND TECHNOLOGY

DEPARTMENT OF PHYSICAL MEASUREMENTS

جامعة. العربي بن مهيدي أم البواقي

معهد العلوم و التقنيات التطبيقية

قســــــم القياسات الفيزيائية

Directed and practical works of

Metrology 2 quality1

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Semester 3 (2024-2025)

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General introduction

This series of tutorials (directed works) and practical works is a logical continuation of the online course already proposed and presented to second-year students in physical measurements at the department of physical measurements of the institute of technology Ain M'lila (Larbi Ben M'hidi University of Oum El Bouaghi). In the tutorial (directed work) part, five series were proposed which give the student the opportunity to understand the metrology subject. The calculations were done following the classical method and the GUM (Guide of Uncertainty Method) recommended by the OECD-Organization for Economic Co-operation and Development. The GUM method is recommended in the program of second year of physical measurement. In the practical part, five series were proposed covering the essential part of the program. The first practical work concerns the calculation of the molar mass of acetic acid with its uncertainty taking into account the different uncertainties related to the atomic masses given by IUPAC. The second practical work represents the calculation of the standard uncertainty related to the glassware; we chose class A and B pipettes and volumetric flasks. In the third practical work, students have the opportunity to calculate the uncertainty related to the slope of a line (from experimental results) using the so-called extreme method using the Excel spreadsheet, the method can be extended to calculating the intercept. The fourth practical work allows calculating the compound uncertainty during an acid-base titration and the accounting of all the errors related to this experiment. Practical work number five relates to the calibration of a measuring instrument, here we have chosen a volumetric flask.

We believe we have understood all the content of the program, periodic improvements will be made to this very important subject, which is metrology. In the descriptive part of this subject, questions and answers according to the skills-based approach are organized during the sessions in order to allow the student to understand the subject well. Many thanks of all my students who, I think without them this work cannot be achieved.

Lecturer
KABOUCHE
Azeddine

Directed Work n°1

Exercise 1

The diameter and mass of a spherical ball are given by:

$d = 10.00 \pm 0.01\ mm$

$m = 9.9 \pm 0.1\ g$

We are asked to calculate:

a- The volume of the ball with its relative and absolute uncertainty

b- The density of the ball with its relative and absolute uncertainty in g/cm3

Exercise 2

We propose to calculate the terrestrial acceleration g using a pendulum. to do this, we measure the length of the pendulum L and the period of oscillation T using the following law:

$$T = 2\pi\left(\frac{L}{g}\right)^{0.5}$$

With $L = 1.552 \pm 0.002\, m$

And $T = 2.50 \pm 0.02\ s$

we ask to calculate:

a-The acceleration g

b- The relative and absolute uncertainty of g

Exercise 3

To measure the height of a building we propose to measure the distance d at which we are as well as the angle under which we see the top of the building

$d = 25.00 \pm 0.01\, m$ and $\alpha = 54^0 \pm 1$

With $H = d \times tg(\alpha)$

Question:

Calculate the height H and its uncertainty

Directed Work n°2

Exercise 1

Let us measure using a stopwatch, the duration t corresponding to 2.5 periods of oscillation of a simple pendulum (5 vertical passages). By having this same measurement made by different students we find:

Test N°	**1**	**2**	**3**	**4**
Duration t	3.62 s	3.47 s	3.44 s	3.30 s

1- Calculate the mean m of the different measurements

2- Calculate the standard uncertainty associated with the measurement of the duration t

3- Calculate the standard deviation of the mean

Exercise 2

1- We want to determine by auto-collimation the focal length of a converging lens. The distance range that allows to obtain the clear image of the object by the mirror is

9.8 cm 11.2 cm

a- Calculate the center of the range

b- Calculate the standard uncertainty

2- We measure a voltage of 4.32 V with a voltmeter on the 20 V caliber, the resolution is 10 mV. The technical notice indicates an accuracy of ± (0.5 % value read + 1 digit).

a- Calculate the error range

b- Calculate the standard uncertainty

Directed work n°3

Exercise 1.

The refractive index of air at 20°C varies with pressure according to Gladstone's law:

$n = 1 + kP$ with **$k = (27 \pm 1).10^{-5}$ bar^{-1}** where **P** is expressed in bar.

Question:

- ✓ What is the value of the air index at 20°C and at pressure **P=2 bar**?
- ✓ What is the value of the uncertainty on this index?

Exercise 2.

We are trying to determine the volume of a steel ball of radius **r= (2.778 ± 0.005) mm**.

The density **$\rho = 7.61 \pm 0.17$ g/cm^3**

Question:

- ✓ Calculate the volume **V** of the ball with its uncertainty
- ✓ Calculate the mass **m** of the ball with its uncertainty

NB: use the GUM method for the second question

Exercise 3.

We consider an electric dipole subjected to the voltage **U= (2.6 ± 0.3) V** and intensity **I= (0.89 ± 0.06) A**. The power is calculated by **P= U.I**

Question:

- ✓ Calculate the electric power **P** supplied to this dipole
- ✓ Calculate its uncertainty

Exercise 4.

In an electrical circuit we measure two voltages **U_1=3.45 V** and **U_2=3.48 V**. In addition, the voltage U has the formula **$U = U_2 - U_1$**. With **$\Delta U_1 = \Delta U_2 = 1$ mV**

Question:

- ✓ Calculate the voltage **U**
- ✓ Calculate the uncertainty

Directed Work n°4

Problem

We are trying to make a life sheet in order to manage a fleet of instruments from a spreadsheet or management software, we then ask to create a table in which we will specify:

1- The name of the instrument (PH meter, conductivity meter, colorimeter, refractometer, etc.)

2- The brand (the manufacturer)

3- The type (the type in this brand)

4- The serial number (that of the device)

5- Inventory number

6- The date of commissioning (date of first use for example)

7- Its allocation (chemistry lab for example)

8- The person in charge (laboratory manager)

9- Type of verification (internal or external)

10- Calibration date (or frequency of calibration of the device)

11- Maintenance (frequency of maintenance)

<table>
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<tr><td colspan="5">Material : Laboratory oven
Brand (mark) : Asus
Type: T6420
Serial number : 44
Inventory number: ....</td><td colspan="4">Date of commissioning : 01/01/2020
Assignment: laboratory of chemistry
Responsible : Laboratory responsible</td></tr>
<tr><td colspan="9">Verification /calibration /maintenance</td></tr>
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Internal ☐
External ☐</td><td colspan="4">Reference procedure : practical guide
Maximum permissible error : ± 5 ° C
Field of use : 105 ° C</td><td colspan="2">Responsible:
Substitute :</td></tr>
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Directed Work n°5

Exercise 1.

We want to calculate the atomic masses and uncertainties of potassium phthalate KHP ($C_8H_5O_4K$).

The atomic mass values are given by IUPAC with a rectangular distribution ($u(x) = x/\sqrt{3}$) for the calculation of the uncertainty. ***MTD*** *: minimum tolerated deviation*

H : 1.00794 g/mole with x=***MTD***= $7.00000\ 10^{-5}$

C : 12.0107 g/mole with x=***MTD***= $8.00000\ 10^{-4}$

O : 15.9994 g/mole with x=***MTD***= $3.00000\ 10^{-4}$

K : 39.0983 g/mole with x=***MTD***= $1.00000\ 10^{-4}$

1. Calculate the molar mass of KHP
2. Calculate the standard uncertainty

Exercise 2.

When calibrating a sodium hydroxide solution (NaOH) with potassium phthalate KHP, the following table is obtained :

	Description	*Value of x*	*standard uncertainty u(x)*	*relative standard uncertainty u(x)/x*
rep	repeatability	1.0	0.0005	……………..
$\mathbf{m}_{KHP}$	weight	0.3888 g	0.00013 g	……………..
$\mathbf{P}_{KHP}$	purity	1.0	0.00029	……………..
$\mathbf{M}_{KHP}$	molar mass	204.2212 g/mol	0.0038 g/mol	…………….
$\mathbf{V}_T$	Volume of NaOH	18.64 mL	0.013 mL	……………..

We are trying to calculate the standard uncertainty of the sodium hydroxide concentration C_{NaOH}

$$C_{NaOH} = \frac{1000 \times m_{KHP} \times P_{KHP}}{M_{KHP} \times V_T}$$

1. Calculate the sodium hydroxide concentration
2. Calculate the standard uncertainty

Practical Work n°1

Estimation of measurement uncertainty

I. REMINDERS:

Error concept
Mesurand *X* : quantity that we want to measure or quantity subject to measurement.
Measurement : set of operations making it possible to obtain a value of the measurand X
Repeatability conditions: the different measurements of X are carried out under strictly identical conditions (same protocol, same instruments, same experimenter, same titrating solution, same day, same place, etc.).
If we repeat (repeatability conditions) the same measurement N times, the results obtained are generally different. We will denote xi the result of a measurement and $\overline{x}$ the arithmetic mean of the N results obtained $\overline{x} = \sum_{i=1}^{N} \frac{x_i}{N}$

Reproducibility conditions: the different measurements of X are not carried out under repeatable conditions. At least one of the following conditions differs: measuring principle, measuring method, observer, measuring instrument, reference standard, location, conditions of use, time. This is the case when the same titration is carried out by different students.
ER measurement error: ***ER = xi - Xtrue***, where ***Xtrue is the real value* of** *X. Xtrue is*, by principle, unknown. In practical work, However, we have a "tabulated value" or "reference value" of X. The ER error is the sum of two contributions: ***ER = (ER)a + (ER)s***
Random error *(ER)a* : ***(ER)a = xi -*** $\overline{x}$. It is caused by the many uncontrollable parameters of the different measurement operations. It can be reduced by increasing the number of observations.
Systematic error *(ER)s* : ***(ER)s =*** $\overline{x}$ ***- Xtrue***. It can be due to the instrumentation (poor calibration of an instrument), to the method (identifying equivalence). It always acts in the same direction. Difficult to detect, it can be corrected.
Accuracy (or exactitude): it characterizes the measuring operations or the instruments. An instrument is all the more accurate as the results obtained under repeatability conditions are close to Xtrue. A correct instrument gives low systematic errors.
Fidelity (or precision): An instrument is all the more faithful as the results obtained under repeatability conditions are close to each other. A faithful instrument gives low random errors.

Uncertainty
Uncertainty, standard-uncertainty *u(x)* : parameter associated with the measurement result, which characterizes the dispersion of the values that could reasonably be attributed to the measurand X. When the parameter used is a standard deviation, we speak of standard uncertainty, noted u(x). The variance is the square of the standard uncertainty. In the absence

of systematic error, the uncertainty defines an interval around the measured value which includes Xtrue.

Composite standard-uncertainty uc(x): standard-deviation obtained by combining all the components of the uncertainty using the law of propagation of uncertainties. It is the square root of the total variance of all the components.
Confidence level: probability of obtaining a result x in the given uncertainty interval.
Expanded standard uncertainty U(x), coverage coefficient k: the expanded standard uncertainty is obtained by multiplying uc(x) by a coverage coefficient that depends on the desired confidence level. Generally, k = 2 at the 95% confidence level.
Presentation of the result of a measurement: X = x U(x) (confidence level). The rounding of x takes into account the value of U(x), which can be given with one or two significant figures. Intermediate calculations are done without rounding.

II. REQUIRED WORK

------ Assessment of uncertainty in the molar mass of acetic acid -----

Atomic mass values are given by IUPAC with a rectangular distribution ($u(x)=x/\sqrt{3}$) for the calculation of uncertainty. *MTD* : Minimum Tolerated Deviation.

H : 1.00794 g/mole with x= *MTD* = $7.00000\ 10^{-5}$

C : 12.0107 g/mole with x= *MTD* = $8.00000\ 10^{-4}$

O : 15.9994 g/mole with x= *MTD* = $3.00000\ 10^{-4}$

QUESTIONS

1- Establish the balance of uncertainty on the molar mass (using the EXCEL spreadsheet)
2- Give the exact value of the molar mass

	Atomic mass	Distribution	Value of x	u(x)	$u(x)^2$
H 4H	-- --				
C 2C	-- --				
O 2O	-- --				
M(CH3COOH)					

Practical Work n°2

Standard Uncertainties Associated with Glassware

I.INTRODUCTION

In this practical work we seek to calculate the uncertainty of some instruments involved in volume measurement in chemistry laboratories. We then propose to calculate the uncertainties of pipettes as well as those of flasks of different volumes.
We consider a triangular distribution with respect to the volume reading on the gauge line for both types of glassware.
We assume a rectangular distribution on the temperature by taking $\Delta T = 4°C$, the expansion coefficient $\alpha = 2.110^{-4}\,{}^{0}C^{-1}$.

$$\sigma_V = \sqrt{\left(\frac{Tol}{\sqrt{6}}\right)^2 + \left(\frac{V.\alpha.\Delta T}{\sqrt{3}}\right)^2}$$

This formula will be applied for the two types of instruments chosen in this PW. Tol: represents the tolerance of each instrument used, it is given by the manufacturer and can be read on the glassware concerned.
The working method in this PW consists of using all the information to calculate the uncertainties of the instruments used using the EXCEL tool or spreadsheet.

II. WORK REQUIRED

QUESTIONS

1- Using the EXCEL spreadsheet, we propose to fill in the following table relating to graduated pipettes (class AS and class B) of different volumes by calculating the absolute errors and the relative errors for each type of glassware:

AS PIPETTES CLASS

V (mL)	1	2	5	10	25	50	100
Tol	0,007	0,01	0,015	0,02	0,03	0,05	0,08
σ_V							
σ_V/V							

B PIPETTES CLASS

V (mL)	1	2	5	10	25	50	100
Tol	0,01	0,02	0,05	0,1	0,25	0,5	1
σ_V							
σ_V/V							

2- Using the EXCEL spreadsheet, we are asked to fill in the following table relating to volumetric flasks of different volumes by calculating the relative error for this type of glassware:

CLASS A GAUGEED FLASKS

V (mL)	25	50	100	200	250	500	1000
Tol	0,04	0,06	0,1	0,15	0,15	0,25	0,4
σ_V							
σ_V/V							

3- Calculate the relative error of a 10 ml AS class burette with a rectangular distribution for the tolerance (0.03 ml) on the volume; a rectangular distribution for the reading with a resolution of 0.05 ml (zero reading plus measurement); a rectangular distribution on the operating temperature at + or – 4 °C compared to the calibration temperature (coefficient of expansion of water 2.1 10^{-4} °C)
4- Calculate the relative error of a 25 ml and 50 ml class B burette for the same data as the previous question.
5- What is the difference between class AS and class B glassware?
6- We propose to calculate the error of an analytical balance to 1/10 of a milligram, for that two mass readings are carried out with a resolution of 10^{-4} g. The maximum permissible error (MPE) of the balance is 0.05 mg (given by the manufacturer)

Practical Work n°3

Calculation of the slope with the extreme method using Excel

METHOD

When we have to calculate an experimental value based on the slope of one side on a graph, the uncertainty about that value is associated with the uncertainty of the slope itself. The location of the points on the graph that have an uncertainty, the slope. The uncertainty will also be present to the right of the trend. Let's examine this uncertainty using a concrete case. Any data series, consisting of x and y coordinates, allows the creation of a graph showing a trend line.

The uncertainty domains of each point can be visualized on the resulting graph. For each point, we can refer to an "uncertainty rectangle", whose uncertainty bars determine the height and width. Generally, the trend line should affect the uncertainty circle at all points. Otherwise, a point could be judged to be erroneous and it would therefore be wise to confirm the measurements of this point if possible.

Step 1

It is possible to use only the first and last points to plot lines of minimum and maximum slope. Since these points may not correspond to the line, the first step is to move them vertically toward the main line. This involves establishing the coordinates of the points on the line located at x relative to the first and last points of the data series used (the same x positions, transposed vertically on the trend line). Insert successively the x coordinates of the first and last points using the equation of the best line (the trend line).

Step 2

Create the uncertainty rectangles corresponding to the first and last points around the new points. We will only use the upper left and lower right corners of these two rectangles (for a positive tilt to the right).

Step 3

Include in the graph the two data series that will connect the top left corner of a rectangle to its bottom right corner. These pairs of points are shown as separate marks on the graph. To incorporate these pairs of points, you can create mini 2x2 tables in your spreadsheet (as opposed to the main table) and enter the x and y coordinates to determine the minimum and maximum slopes.

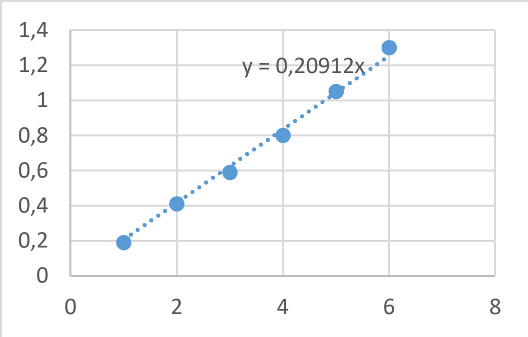
Step 4

Show the trend lines of these two pairs of points and request the presentation of their equations. The uncertainty will be calculated using the extreme values of the slope (a_{max} and a_{min}) of these two new lines, as shown in the example.

Calculating of min and max slopes

Calculating the tendency curve

X	Y	ΔX	ΔY
1	0,19	0,05	0,02
2	0,41	0,05	0,02
3	0,59	0,05	0,02
4	0,8	0,05	0,02
5	1,05	0,05	0,02
6	1,3	0,05	0,02



We obtain the slope from the tendency curve

$slope = 0.20912$

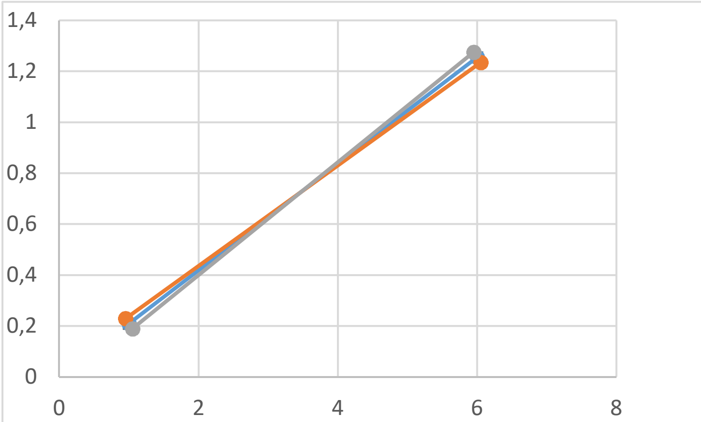
The curve passes through zero

Creating the line 1-2

Point 1	**1**	**0,20912**
Point 2	6	1,25472

$x_1 = 1$ $\qquad$ $x_2 = 6$

$y_1 = slope \times x_1$ $\qquad$ $y_2 = slope \times x_2$



Creating the line A-D

Point A	**0,95**	**0,22912**
Point D	6,05	1,23472

$x_A = x_1 - \Delta x$ $\qquad$ $x_D = x_2 + \Delta x$

$y_A = y_1 + \Delta y$ $\qquad$ $y_D = y_2 - \Delta y$

Creating the line B-C

Point B	**1,05**	**0,18912**
Point C	5,95	1,27472

$x_B = x_1 + \Delta x$ $\qquad$ $x_C = x_2 - \Delta x$

$y_B = y_1 - \Delta y$ $\qquad$ $y_C = y_2 + \Delta y$

Calculating slope AD

$$a_{AD} = \frac{y_D - y_A}{x_D - x_A} = 0.1972$$

Calculating slope BC

$$a_{BC} = \frac{y_C - y_B}{x_C - x_B} = 0.2216$$

Calculating the mean slope

$$a_{mean} = \left|\frac{a_{\max} + a_{\min}}{2}\right| = 0.2094, \quad \Delta a = \left|\frac{a_{\max} - a_{\min}}{2}\right| = 0.0122$$

Result presentation

$a = 0.2094 \pm 0.0122$

Question :

Use Excel to reproduce this practical work following the steps and using the values in the table above

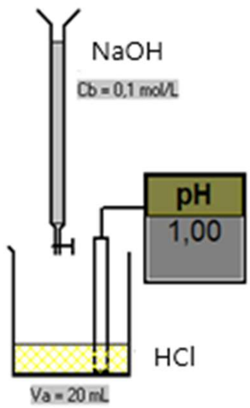
Practical work n° 4

Calculation of the composed uncertainty during a strong acid titration by a strong base

I. INTRODUCTION

The objective of the PW is to calculate the compound uncertainty when dosing hydrochloric acid (HCl) with soda (NaOH). This involves counting the various errors made during this dosage, we can cite:

- Error due to the repeatability of the experiment
- Error due to the temperature because of the expansion of the liquids
- Error due to visual detection (using the colored indicator, the error is systematic)
- Maximum Tolerated Deviation (MTD) given by the manufacturer (here we use 25 mL burette)



The experimental protocol is that of last year in the chemical analysis module, in particular in the practical work entitled: dosage of a strong acid by a strong base and the practical work on pH-metric titrations. It is therefore a question of determining the concentration of the acid while knowing that of the base but this time in an exact manner, it is therefore necessary to have the necessary error and uncertainties.

The results of five dosages (repeatability) are given in the following table:

Table 1

Acid-base dosage table					
EXP	**1**	**2**	**3**	**4**	**5**
Veq(NaOH mL)	**20,2**	**20,2**	**20,1**	**20,4**	**20,3**
Veqmean (mL)	**?**				
Standard uncertainty	**?**				

For the calculation of the combined uncertainty we use the following table:

Table 2

Error Sum Table					
	Formula	**Distribution**	**Value of X**	**u(x)**	**u(x)^2**
Temperature	2,1E-4*dt*Veq	Rectangular	**?**	**?**	**?**
Constructor	*MTD*=0,5%*Vtotal	triangular	**?**	**?**	**?**
Repeatability		Normal	?	**?**	**?**
Visual Detection		Normal		**0,00003**	**?**
dt=2,6				**SumErr=**	**?**
				Error =	**?**

Question

1- Calculate the average equivalent volume
2- Calculate the standard uncertainty on the average
3- Find the composite error based on the information in the previous tables.
4- What is the predominant component?

Practical work n° 5

Calibrating a measurement instrument

I. INTRODUCTION

The result of a measurement is never a value: it will be given in the form of an interval of probable values of the measurand M = m ΔM associated with a confidence level. A significant part of the experimental work therefore lies in the estimation of M, called the confidence interval associated with a given confidence level. When uncertainties are evaluated by statistical methods, the evaluation is said to be type A. When statistical determination is not possible, the evaluation is said to be type B. When the sources of variability of the measurement are multiple, the standard uncertainty is estimated for each of them and an overall assessment is made to construct a composite standard uncertainty, which can mix type A and type B evaluations.

1. Type A assessment of standard uncertainty

Type A assessment of standard uncertainty is carried out by statistical analysis of series of observations (GUM). If we assume the n observations mk are independent.

- The best estimate of the measurement result is given by the arithmetic mean:

$$m = \overline{m} = \frac{1}{n}\sum_{k=1}^{n} m_k$$

- The experimental standard deviation is expressed as :

$$s = \sqrt{\frac{1}{n}}\, s_{exp}$$

2. Type B assessment of standard uncertainty

Type B assessment is performed by means other than statistical analysis of series of observations. For an estimate of an input quantity that has not been obtained from repeated observations, the standard uncertainty is assessed by scientific judgement based on all available information about the possible variability of the input quantity (GUM). The accumulated body of information may include:

- Previous measurements;
- Experience or general knowledge of the behaviour and properties of the materials and instruments used;
- Manufacturer's specifications;
- Data provided by calibration certificates or other certificates;
- Uncertainty assigned to reference values from a book or manual.

II. EXPERIMENTAL PROTOCOL

We seek to carry out a type A assessment of this uncertainty in relation to glassware used in a chemistry lab. We will use a flask with known precision (here we check whether our value coincides with that of the manufacturer) and a graduated test tube without precision.

Principle of determination:

Deliver N times the volume V and check by weighing the volume delivered from the density of water at the measurement temperature. In our experiment: N = 5; V = 20 mL (or 25 mL) for the flask (fiole) and N=5 and V=10 mL for the test tube.

Test	MassGlassware (g)	MassGlassware+H_2O (g)	T °C	ρ_{H2O} in (g/L)	GlasswareVolume (mL)
1					
2					
3					
4					
5					

Questions :

1. Fill in the previous table
2. Calculate the average of the volume measurements
3. Calculate the standard deviation on the volume
4. Propose another measurement method
5. Compare with the manufacturer's data

Nota Bene: The density of water as a function of T is given by the following table:

T(°C)	15	16	17	18	19	20	21
ρ_{H2O}(g/mL)	0,999099	0,998943	0,998775	0,998596	0,998406	0,998205	0,997994
T(°C)	**22**	**23**	**24**	**25**	**26**	**27**	**28**
ρ_{H2O}(g/mL	0,997772	0,99754	0,997299	0,997047	0,996785	0,996515	0,996235

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