General Lab Manual

Guide for performing experiments

Advanced Technology

Chemical Science & Engineering

Technische Natuurkunde

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Authors:

Michel de Jong, Hans Kanger, Carlijn van Emmerik, Jéré van Lente, Cornelise Vreman, Jasper Homminga, Sander Wildeman, Aernout van Rossum

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1. Role of experiments in natural sciences

By the end of this chapter, you should be able to mention a couple of reasons why one would do experiments in chemistry, physics or any other natural science.

Experiments are the primary method for testing hypotheses and validating theories. Experiments can also call for a new theory, either by showing that an accepted theory is incorrect, or by exhibiting a new phenomenon, which needs explanation (Franklin, 2007).

1.1. Testing Hypotheses

Formulation of hypothesis and performing controlled experiments Scientists begin with a hypothesis, a tentative explanation or prediction that can be tested through experimentation. By manipulating one or more variables while keeping others constant, scientists can determine the effects of these variables on the outcome. An early example of a hypothesis and controlled experiments is performed by Galileo who wanted to test whether objects of different masses fall at the same rate.

Galileo (156401642), who could link theory with experiment

- **Objective**: Galileo was curious to see how things fell toward the earth. However, he could not measure free falling motion and time, as technology was unable to record such high speeds. He tried to decelerate motion by rolling balls down an inclined plane.
- **Setup**: He designed an inclined plane on which balls could roll down.
- **Procedure**: He observed that balls of any weight rolled toward the earth at the same rate. Later this constant was called gravitational acceleration.
- **Result**: This experiment supported the idea that gravitational acceleration is independent of an object's mass.

Figure 1 Experimental device of Galileo Galilei for experiments of the law of falling bodies. In rolling down the inclined plane, the ball hits the bells which are attached following a quadratic law. This device is rebuilt in Florence. (Frauenberger, 2007)

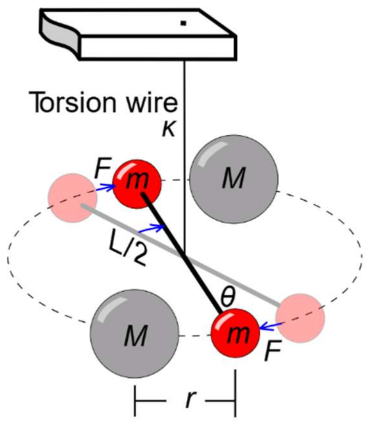
Please note:
Galileo is also known for his development and use of scientific instruments and mathematics. He developed methods to measure short periods of time more accurately and he improved the telescope design.

1.2. Validation and extension of Theories:

Empirical evidence is used to support or refute a theory and build on a theory. For a theory to be widely accepted, experiments must be reproducible by other scientists under the same conditions. Scientists can think of experiments that extent a theory building on what is already validated. Building on the gravitational theory Henry Cavendish performed experiments to determine the gravitational constant.

Cavendish Experiment (1797-1798) Weighing of the earth

- **Objective**: Cavendish was interested to know whether objects would attract each other. Therefore, he wanted to measure the force of gravitational attraction between masses and determine the gravitational constant (G).
- **Setup**: Henry Cavendish used a torsion balance apparatus consisting of a horizontal bar suspended from a thin wire. Small lead spheres were attached to the ends of the bar, and larger lead spheres were placed nearby.
- **Procedure**: The gravitational attraction between the small and large spheres caused the bar to rotate, twisting the wire. The angle of rotation was measured to calculate the force of attraction.
- **Result**: Cavendish's experiment provided the first accurate value for the gravitational constant, allowing the calculation of the Earth's mass.



A nice movie showing the experiment can be found here: [Gravitational Attraction Cavendish experiment]

Figure 2 Diagram of the torsion balance used in the Cavendish experiment (Burks, 2007)

Please note: In fact, the measurement procedure was more complicated. One problem was that the torsion balance would not sit still, but would keep oscillating. So, he actually recorded the amplitude and period of the sinusoidal motion. In addition, he had to make sure other factors like static electrical charges or wind in the room were not at play. His measurements and error analysis have been excellent. Researchers after Cavendish have not been able to improve his accuracy.

1.3. Discovery of New Phenomena:

Sometimes experiments yield unexpected results, leading to the discovery of new phenomena or the development of new theories. Experiments can be designed to explore unknown aspects of the natural world, leading to new insights and knowledge.

Discovery of polonium and radium

Objective: Around 1896, it was discovered that uranium salts emitted rays that resembled X-rays. Madame Curie decided to look into uranium rays as a possible field for a thesis and she was interested in finding methods for the treatment of neoplasms by the use of radioactive isotopes.

Procedure: She used an electrometer, a sensitive device for measuring electric charge, to investigate samples.

Results: Activity of uranium compounds depended only on the quantity of uranium present. She hypothesized that the radiation was not the outcome of some interaction of molecules, but had to come from the atom itself.

This work laid the foundation for the field of nuclear physics and had profound implications for medicine, particularly in cancer treatment.

A nice overview of their work, can be found at the website of the PSL university of Paris (PSL, 2025).

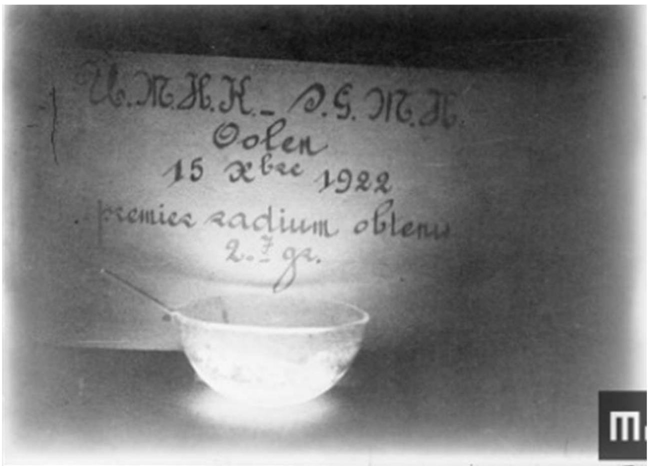


Figure 3 A bowl containing radium bromide (photo taken in the dark) (PSL, 2025).

1.4. Educational value of experiments

Within CSE, TN and AT experiments have an educational value as they help students understand scientific concepts and provide a familiarity with apparatus and equipment. In addition, conducting experiments provides hands-on experiences in systematically performing experiments.

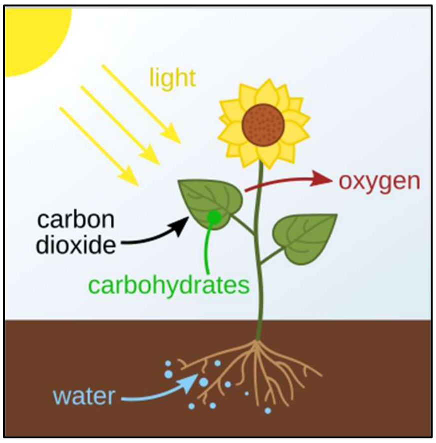
Summary: Experiments are the backbone of the natural sciences: they provide a systematic way to investigate the world, test ideas, and build a body of reliable and interconnected knowledge. Experiments are essential for the advancement of technology and vice versa: Advancements in technology is essential in the advancement of experimental science. Often, developments in technology and experiments go hand in hand.

2. The Scientific Method

By the end of this section, you should be able to explain the basic steps of the scientific method.

Within the natural sciences, a systematic approach is used by scientists to explore observations, answer questions, and test hypotheses. This structured process ensures that scientific investigations are objective, consistent, and replicable. The following example from biology shows the main steps of the scientific method (Wikipedia, 2025).

2.1. Application of the Scientific method

1. **Observation & question**: A gardener notices that plants in a shaded area grow slower than those in direct sunlight.


Does sunlight affect the growth rate of plants?

2. **Research**: The gardener reads about photosynthesis and plant growth. See figure 1.

3. **Hypothesis**: If plants receive more sunlight, then they will grow faster.

4. **Test with experiment**: Now the gardener comes up with a method to test the hypothesis. The gardener plants two groups of identical plants, one in direct sunlight and one in the shade, and measures their growth over a month.

5. **Data Analysis**: The gardener compares the growth rates of the two groups. The gardener represents the data in a graph. From this graph the gardener can draw a conclusion.

6. **Reporting and conclusion**: The plants in direct sunlight grew faster, supporting the hypothesis. Other gardeners or scientists might replicate the experiment to confirm the findings.

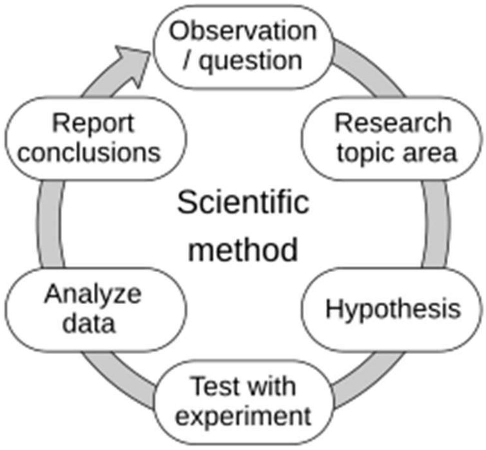
Continuing research: The gardener might want to investigate whether different wavelengths of light have different effects on growth of plants.

Please note: This example is over simplified, the depiction in steps is to give you an overview of stages in research. In practice, however, the research process is more complicated and less linear. Of course, other aspects (e.g., plants are not really identical) might play a role as well.

2.2. General steps in the Scientific Method

Although procedures vary across disciplines, the underlying process is often similar. General steps in the scientific method are the following:

1. **Initial observations and question:** The process begins with observing a phenomenon or a set of phenomena or a question one has about a theory. These observations can be based on anything from natural occurrences to experimental results. From these observations, a specific question is formulated. This question aims to address the observed phenomena.
2. **Research:** Before forming a hypothesis, scientists conduct background research to understand what is already known about the topic. This helps in refining the question and designing the experiment.
3. **Formulation of hypothesis**: A hypothesis is a suggested explanation or prediction of the phenomenon. The hypothesis can be tested through experimentation. It is often stated in an "if-then" format (e.g., "If I water plants more frequently, then they will grow faster").
4. **Designing and conducting the experiment**: An experiment is designed to test the hypothesis. This involves identifying variables, creating a procedure, and determining how data will be collected.



5. **Data Analysis**: The collected data is analysed to determine whether it supports or refutes the hypothesis. This can involve statistical analysis, graphing, and other methods of data interpretation.
6. **Drawing conclusion and reporting results:** Based on the data analysis, a conclusion is drawn. The conclusion states whether the hypothesis was supported or not. The results are often shared with the scientific community through publications, presentations, or reports.

One could continue research by e.g.,

- **Replication and Peer Review:** Other scientists may replicate the experiment to verify the results. Replication is crucial for establishing the reliability and validity of the findings. Before publication, the research is typically reviewed by other experts in the field to ensure the methodology and conclusions are sound.
- **Theory Development:** If a hypothesis is repeatedly supported by many experiments, it may contribute to the development of a scientific theory. A theory is a well-substantiated explanation of some aspect of the natural world that is based on a body of evidence.

Although the above procedures might seem straightforward, experimenting is not a linear process and it requires a surprising amount of creativity and perseverance.

2.3. Application of the scientific method

During your experiments, you will apply this scientific method. In the first labs, the assignments will guide you through the method. Gradually, you will have to define the steps yourself.

3. Safety on the lab

Whenever you are working with physical objects, there is a risk involved. Sometimes this risk is obvious (e.g. you are working with boiling water) or a little more rare but still easily conceptacle (e.g. the equipment you are working with could short-circuit and cause a fire). However, especially when working with chemicals, the dangers are often both more severe and more difficult to conceptualize. Something that burns away your skin is easy enough, but what do you do with something that increases the risk of cancer by X% over Y number of years?

Fortunately, for that exact reason chemists have developed a form of 'safety language' in the form of shared symbols, phrases, and customs. It is incredibly important that everyone on the lab is aware of and abides by safety rules. Not only for yourself, but also for others.

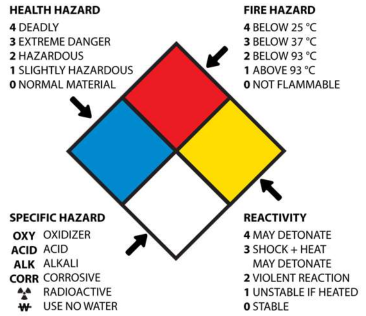
This section is a very short overview of the most important general lab practices and information. But the rule above all others is: **know what you are doing**. Be aware of what chemicals you are using, what their dangers are, what precautions to use, how to dispose of reagents, and what to do in case of spills or worse. Take into account concentrations and amounts as well; you don't need to treat a 5 M NaOH solution the same as a 5 mM NaOH solution.

General rules

- **Wear lab goggles**. Your eyes are sensitive and you only get 1 pair.
- **Wear a lab coat**. The purpose of the lab coat is to act as an extra barrier between you and the chemicals.
- Cover up skin as much as reasonably possible, that means wearing **closed shoes** and **long pants**. Like the lab coat, the purpose is to act as an extra barrier.
- **Never eat and drink** in the lab. Your skin is a reasonably effective barrier against contamination, eating or drinking accidentally contaminated material is a good way to get a trip to the hospital. In addition, you don't want to contaminate your experiment.
- **Prepare** by knowing what you are going to do.
- Wear **gloves when necessary**, and don't wear them when not necessary. Know what types of gloves provide protection against your chemicals.

Quick safety references

- H-statements are small sentences that inform you about the **hazards** of a chemical. Older chemicals may still list ‘R-phrases’. These are essentially the same thing.
- P-statements are small sentences that inform you about the **precautions** you should take when handling/storing a chemical. Older chemicals may still list ‘S-phrases’.
- The **‘safety square’** (also known as ‘**fire diamond**’) is a four-section multicolored square that gives a quick overview of the most important safety aspects of a chemical. See next page.
- A standardized series of red-bordered pictograms are used to represent dangers posed by a certain chemical. The most common are listed on the next page.
- If you **ever** have to use or handle a chemical with pictograms or symbols you do not understand, stop immediately what you are doing and make sure you understand them. I promise you that you will have the time. N**o experiment or assignment is worth a risk to your health**.

<table>
<tr>
<td>

By Marcos Rodríquez Bobabdilla</td>
<td>
Explosive</td>
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Flammable</td>
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<tr>
<td>
Oxidizing / Oxidizer</td>
<td>
Compressed gas / Gas under pressure</td>
<td>
Corrosive / strong acid or base</td>
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<tr>
<td>
Toxic</td>
<td>
Health hazard (but less than toxic) / Hazardous to the ozone layer</td>
<td>
Serious health hazard in a biological way (e.g. carcinogenic or mutagenic)</td>
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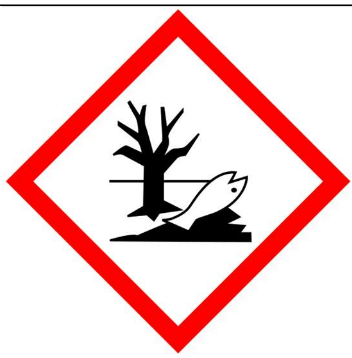
<td>
Hazardous to (aquatic) environment</td>
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</table>

4. Representing data

4.1. Select the best representation for your data

Using tables, graphs and figures in journals and reports allows you to present the data in a compact and structured way. Additionally, in publications they form an important way to access the information provided. It is therefore important to format them according to universal guidelines. As there is a wide range of graphs, figures and tables to represent your data, how to select a representation that provides the best insight into your results? Two important questions can guide you in this process:

1. **What is the goal of your figure?** For example, you want to show differences or similarities between data sets, or show a relationship between variables, or you want to determine the optimum of a variable.
2. **What kind of variables have been used.** For example, you measured the waterflow in a pipe over time (which could be a continuous line), or you measured the number of students passing a course in relation to their high school math grade (as the math grade is rounded to half digits, it is a discontinuous variable).

The most important types of graphs, their purpose and an example are presented in Table 1.

Table 1 Overview of the main types of graphs.

Type of graph	Purpose	Example
Line Graphs	Ideal for showing trends and changes over time or continuous intervals.	Displaying temperature changes over a period, or population growth.
Bar Graphs	Used for comparing data between different categories or groups.	Comparing the prevalence of diseases in different regions.
Scatter Plots	Used to visualize the relationship or correlation between two numerical variables.	Exploring the relationship between height and weight,
Histograms	Show the frequency distribution of numerical data, often used to identify patterns in continuous data.	Displaying the distribution of test scores or the height of individuals in a population.
Pie Charts	Used to display proportions or percentages of a whole, often showing how a whole is divided into parts.	Representing market share of different companies or the distribution of resources in a budget.
Table	Displaying precise and raw data in a structural format.	Time measured for 100 m swimming for each participant.

4.2. Dependent and independent variables

When you measure a physical quantity as a function of some other quantity we speak of a dependent variable and an independent variable.

- The independent variable is the variable you change in the experiment. It is the cause that is believed to have an effect on the dependent variable.
- The dependent variable is the variable that might change depending on the independent variable.

In the experiment of Galileo discussed in chapter 2, one could change the height of the ramp. The independent variable is the height of the ramp. The dependent variable is the time it takes for the ball to roll down.

4.3. Characteristics of a good table

A table is a collection of (raw) data organized in rows and columns. Tables display data precisely and clearly, so you can see the exact numbers you're working with. Tables are used to organize data for analysis, closely examining numbers or making direct comparisons.

Guidelines for making a good table	
Title:	The table has a title above the table. The title explains what data is in the table.
Numbering:	Numbers (usually Roman I, II, III etc) are used to label the table. Use these numbers in the text to cite the table.
Header:	The header of the table lists the symbols of quantities and their units (e.g., *t* [min])
Errors:	The table should also include the errors/uncertainties in the measurements if applicable If the error is the same for all measurements, it is common to put the error in the table caption.

Table I Readings of a radioactive decay experiment. The first column reads the time t (± 0.02 min) and the second column gives the activity $A \pm$ s.d..

t [min.]	A [counts]
0	4483 ± 134
24	2973 ± 109
48	2099 ± 92
72	1501 ± 77
96	1061 ± 65
120	739 ± 54
144	490 ± 44
168	311 ± 35
192	220 ± 30
216	154 ± 25
240	116 ± 22
264	60 ± 15
288	47 ± 14

Figure 4 Example of a table. Taken from the Reader Data analysis (TN).

To illustrate the guidelines, you could have a look at Table I. The data from an experiment measuring activity of a radioactive sample as function of time is presented.

4.4. Roles and characteristics of a good graph

Graphs play an important role in communicating scientific findings in a clear and concise manner. A good graph should be readable (use proper size of text etc.), it should be concise (do not overcomplicate graphs but keep it simple) and it should be understandable (use captions, axis labels, etc to explain what the graph is about).

In addition, graphs play an important role in data analysis as they aid in visualizing relationships and understanding experimental results. For this reason, consider things like: what **scale** suits the purpose of the graph (e.g. plotting on a logarithmic scale can make it easier to check for exponential behaviour) or how to compare data sets (e.g., by plotting multiple datasets in one graph, by plotting the theoretical model on top of the data sets). Figure 5 shows the basic building blocks of a graph and the terminology.

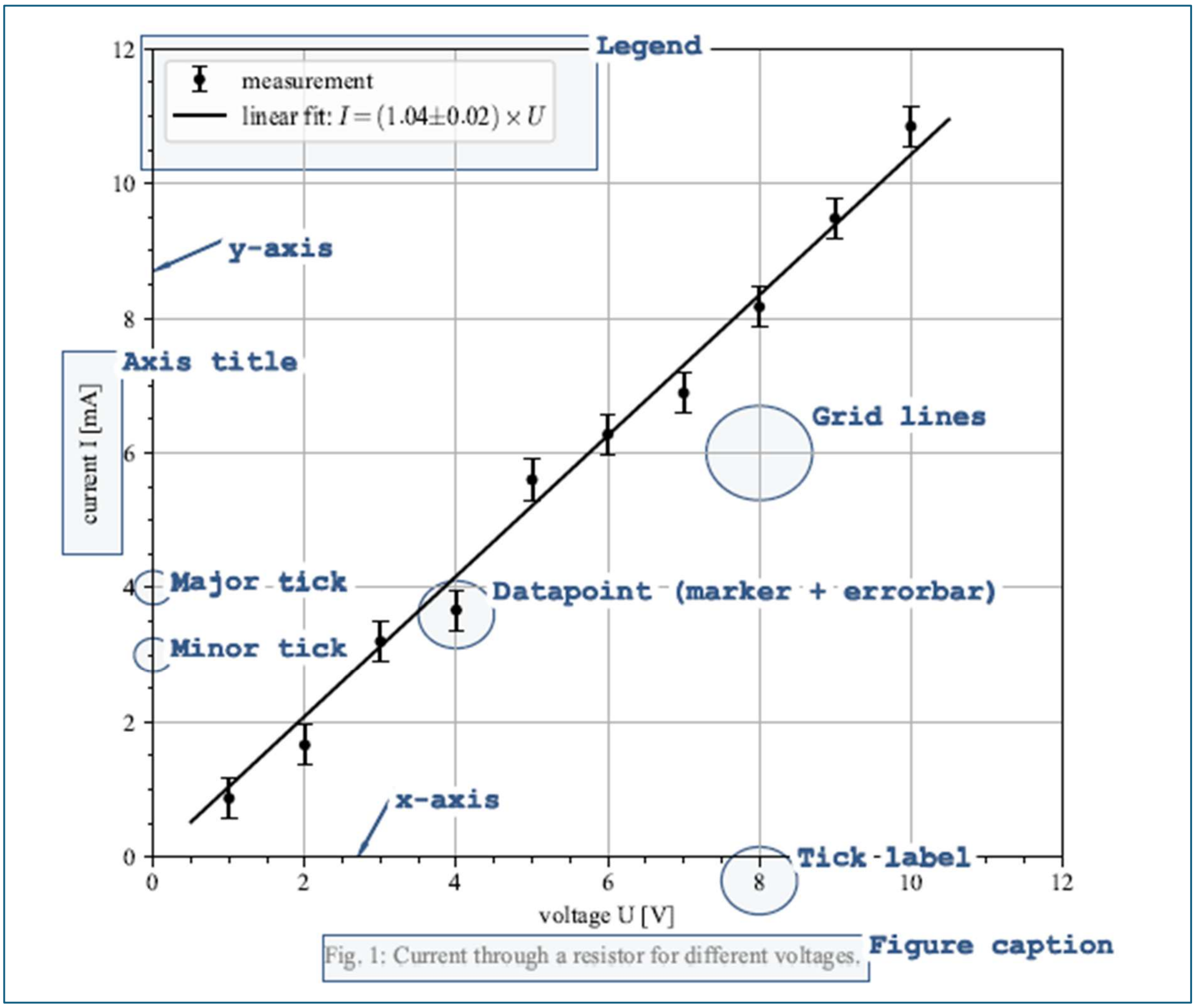


Figure 5 Anatomy of a good graph.

Ten Rules for making a good graph

Identify the variables: The independent variable goes on the x-axis, the dependent variable goes on the y-axis.

Label each axis: Include both quantity and unit and if appropriate the symbol of the the quantity (e.g. Current I [mA])

Axis range: Choose the range of the axis carefully. Use a range such that the data points are distributed nicely over the whole graph.

Markers: Use markers to plot datapoints and do NEVER EVER connect them by a line. In case of multiple datasets in one graph use preferably different markersymbols (□, ◊, ▲) rather then different colors.

Lines: Use a line to show relationships between variables when you have a mathematical function/model (e.g. from a fit). If you have multiple lines use different linestyles (—, $\cdots$, etc) rather then different colors.

Legend: If your graph has multiple datasets and/or lines, a legend provides a key to the different datasets and or lines. Alternatively you use the figure caption for this.

Errorbars: Include errorbars in your graph if applicable.

Size: The graph should be readable. Choose a fontsize for the text in the graph at least as big as the fontsize of the figure caption (usually 10pt). Also linewidths, markersize, etc should be clearly visible when printed (and not only on your computer screen!).

Caption or Title Always include a figure caption that explains what is plotted against what. A suggestion for a proper caption: "The dependence of (your dependent variable) on (your independent variable)". If you are using the graph in a presentation use a title above the graph instead of a caption.

Linearize Very often a graph is used to check if the data follows some theoretical model. In this case it can be wise to use scales (linear, logarithmic, reciprocal) that would (at least theoretical) result in a plot where the datapoints are on a straight line.

5. Error Analysis - overview

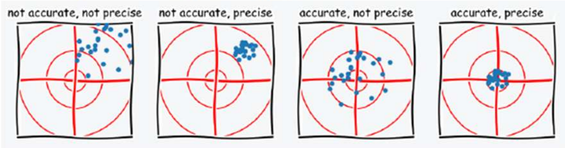
ERROR ANALYSIS the TL;DR version

SOURCES OF ERRORS

Measurement errors in experiments stem from five main sources:
- **Observer**: e.g. reading errors (of scales) or reaction delays.
- **Equipment:** limited resolution, calibration issues.
- **Method:** e.g. assumptions may introduce systematic errors.
- **Environment:** factors like temperature can affect results.
- **Fundamental noise:** e.g. in low signals shot noise is unavoidable.

CLASSIFICATION

- **Systematic errors**: Are the same for each measurement and do not average out through repetition. Determine **accuracy**.
- **Random errors**: Are random in nature and will average out with many repetitions. Determine **precision**.



NOTATION

Measured quantities are written as:

"value ± error [unit]"

- Round the error to **1 significant figure**, preferably **upward**.
- If the error is between **1 and 1.5**, you may use **2 significant figures**.
- Choose the number of significant figures in the value to match the size of the error.

Examples:

g = 9.81 ± 0.03 ms^{-2}
I = (4.21 ± 0.13) × 10^3 Wm^{-2}

You can also express the **relative error** as a percentage:

"value [unit] ± relative error %)"

Example:

g = 9.81 ms^{-2} ± 0.3%

ERROR ESTIMATION

- **External error estimation**:
  When measuring a single value of a quantity, you determine (or estimate) the error using documentation of the equipment used.
  In addition, you estimate errors such as reading errors from the scale (for analog scales) or consider the least significant digit (for digital displays).
- **Internal error estimation**:
  You estimate the measurement error by looking at the **spread of values** when repeating the same measurement multiple times.
  (See separate section on internal error estimation.)
  Note: This method only yields the **statistical (random) error**, **not** the **systematic error**!

INTERNAL ERROR ESTIMATION

In an **internal error estimation**, you make use of the **random nature of the error**.

1. Take a **sample** of **N measurements** :$x_1, x_2, x_i \ldots x_N$.
2. Calculate the **mean**:

$$\bar{x} = \frac{1}{N}\sum_{i=1}^{N} x_i$$

3. Calculate the **unbiased standard deviation** (*sample standard deviation*):

$$\sigma_s = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}(x_i - \bar{x})^2}$$

σ_s gives the **estimated error in a single measurement**.

CONFIDENCE INTERVALS (CI)

Confidence interval (CI) (or **margin of error**) indicates how reliable the result is. The **confidence interval** of the sample mean is given by:

$$\pm t\frac{\sigma_s}{\sqrt{N}}$$

•For **N > 25**: **t ≈ 1** for a **68% confidence interval**
t ≈ 2 for a **95% confidence interval**

•For **N < 25**: Determine **t** from a **Student's t-distribution table**

Interpretation: A **95% confidence interval** means that there is a **95% chance** the interval contains the **true value** of the measured quantity.

PROPAGATION

- Functions $f(x)$ of a single variable x:

$$\delta f = \left|\frac{df}{dx}\right|\delta x$$

- Functions $g(x,y,..)$ of multiple variables x, y,.., where the errors in the variables are **independent** and **random** in nature:

$$(\delta g)^2 = \left(\frac{\partial g}{\partial x}\right)^2 (\delta x)^2 + \left(\frac{\partial g}{\partial y}\right)^2 (\delta y)^2 + ..$$

This is the standard formula for **propagation of uncertainty**.

COMPARISON OF RESULTS

When comparing an experimentally obtained value with another (expected or known) value, it is common to report the following:
- Results **agree well (excellent agreement)** if the expected value lies within the **68% confidence interval**.
- Results **reasonably agree** if the expected value lies within the **95% confidence interval**.
- Results **do not agree** if the expected value lies **outside the 95% confidence interval**.

FITTING

Purpose**: validation** of a theoretical model or **determination** of unknown parameter(s). Given **N data points** (x_i, y_i) and a model function $y(x, p_1, p_2,..)$ where $p_1, p_2,$ are the fitparameters, the **least-squares** method estimates p_1, p_2.. By minimizing the sum S:

$$S = \sum_{i=1}^{N} w_i(y(x_i, p_1, p_2, ..) - y_i)^2$$

where w_i is the **weight** of each data point, typically $(1/\delta y_i)^2$.

Criteria for a good fit:
- Check for **outliers** and remeasure if necessary
- Fit **only the function** specified by the model
- **Linearize** the model if possible
- Check whether the **residuals** of the fit are **random** (no trend or pattern)

PYTHON CODE

```
import numpy as np
from scipy import stats

data = np.array([x1, x2, x3, ...])
N = len(data)
```

```
mean = np.mean(data)
std_dev = np.std(data, ddof=1)
confidence = 0.95
t_value = stats.t.ppf((1 + confidence) / 2, df=N-1)
CI = t_value * std_dev / np.sqrt(N)
```

6. Documenting experiments in a lab journal

6.1. Introduction

Documenting experiments is a crucial part of scientific research. Initially, one typically records the preparation of the experiment, observations and results during the experiment, and (preliminary) conclusions in a lab journal. This journal subsequently serves as the backbone to summarize key findings in a more formal research report. These methods of documentation help to ensure that valuable data is not lost, allow other researchers to replicate the experiments under the same conditions, and provide a basis for follow-up studies.

> ⚠ **Important**
>
> It should be possible to **reproduce** the experiment (including results and analysis) **exactly** based on your journal.

This chapter explains the importance of maintaining a lab journal and outlines the methodology for creating one effectively.

6.2. Structure of lab journal

A lab journal is developed in three stages:

1. **Preparation** In this phase, the objective of the experiment is defined, an expectation is formulated, and the measurement model and procedure are outlined. This step lays the foundation for a structured execution.

2. **Execution** During this phase, the experiment is carried out. All observations, measurements, and relevant conditions are carefully documented and explained where necessary.

3. **Reflection** In the final phase, the results are analysed and the accuracy of the measurements is assessed. The findings are discussed, conclusions are drawn, and recommendations are made for improving the experiment.

The most important items for a lab journal are explained in the **standard lab journal**. Always be aware that you document only the parts that are relevant to your experiment!

As far as the structure is concerned, there is only one condition, which results from the logical structure of the experiment and the chronological character of the journal. Every experiment has to be prepared (at home) and every journal will therefore start with the documentation of this preparation. After that, the description of the execution of the experiment and the processing of the measurements follows and based on the results the conclusions are drawn. As a result, the journal will reflect this structure automatically.

6.3. The standard journal structure

The standard journal structure given below provides a guideline for keeping a journal. In the long run most journals will deviate from this example and experimenters will develop their own style. But this structure is a good starting-point. As said before: parts that are not relevant for a specific assignment are left out.

Preparation	title	First, write the assignment number and title of the experiment
	collaboration	If the work is done in collaboration, provide the name of the partner
	date	State the date, and if relevant the time
	<u>Objective of the experiment</u>	
	goal	From the description of the assignment, determine and briefly formulate a specific goal. Note: The title of the assignment is not the goal.
	expectation	Which specific results do you expect? This could be a numerical value for the quantity to be measured, a graph, material properties, or specific reactions. If applicable refer to relevant theory or literature.
	<u>Measurement method</u>	
	measurement model	The measurement model describes the relationship between the quantity of interest (the measurand) and the quantities that are measured directly during the experiment. It provides the theoretical basis for converting the measured data into the desired result. The measurement model (usually) includes: • A clear schematic of the measurement setup, showing the relevant components, the directly measured quantities, the measurand, and the instruments used to obtain each measurement. • A measurement equation, derived or justified from the underlying theory, that expresses the measurand in terms of the directly measured quantities. Include the essential derivation steps, state any assumptions, and clearly define all variables, constants, and their units. For example, to determine the temperature of a substance using a thermocouple, the temperature needs to be expressed in terms of the measured thermocouple voltage. The meaning of the variables (in relation to

	the experiment) should be made clear.
data preparation	Describe how will the collected data will be processed in practice to apply that measurement model described above. Indicate the analysis steps that will be performed after the measurements have been collected. For example, specify whether the data will be substituted directly into the measurement equation, averaged, corrected using calibration data, fitted to a model, or analysed graphically. • If graphical analysis is used, specify what is plotted on the x-axis and y-axis (including units), • the purpose of the graph (e.g., determining a slope or intercept), • whether linear, logarithmic, or another type of scale will be used, • any transformations or calculations applied before plotting.
error analysis	Discuss for every quantity to be measured • the nature of the measurement error(s) (where does the error come from? will it be a random or a systematic effect?), • how you could keep the error(s) as small as possible (using the available equipment), • how you plan to *estimate* the errors during the experiment. Next, derive error propagation formulas for the quantities that will be calculated from the measured values (as specified in the measurement model). These formulas should be developed up to the point where the uncertainties in the calculated quantities are expressed in terms of the errors of the measured values. <u>Note</u>: The actual numerical errors can only be determined during the experiment. Therefore, calculating the specific error values belongs in the *Results* section, as part of the error processing.
<u>**Experimental**</u>	
measurement procedure	Describe as precise as possible your procedure (including equipment settings etc) during the experiment. Keep, in a different colour, track of any important changes you make along the way (and the motivation behind these changes). In principle, the procedure should be explained in enough detail such that someone else could reproduce the

		experiment (and your results) without additional clarification.
	safety	Specify any safety regulations that must be followed to prevent dangerous situations. For chemistry experiments: Gather safety information for all chemicals involved in the experiment. Translate these to concrete measures for the situation at the lab: what precautions should you take and what to do in case of spilling, skin or eye contact, inhalation etc. Take into account the quantities and concentrations used. You do not need to treat 1 M HCl the same as 1 μM of HCl. Clearly indicate how chemical waste should be disposed of, including the appropriate waste category for each substance. For physics experiments: Consider safety measures to prevent damage to equipment and ensure safe operation. This may include proper grounding, avoiding overloading circuits, using shielding, and following manufacturer guidelines for sensitive instruments.
Execution	setup	Create a detailed sketch of your experimental setup. Provide name, type and code number for each piece of equipment/device. Specify the settings of the equipment and how it was calibrated (if appropriate). You could also include photos of important details of your setup. Make sure to annotate your sketches/photos clearly.
	Results	
	measurements and observations	Record raw measurement data clearly using tables, graphs, or photos as appropriate and include estimates of errors. Also include relevant (qualitative) observations that may explain unexpected behaviour or influence the interpretation of the measurements. Examples include vibrations, unstable readings, environmental conditions, equipment malfunction, etc. If data is stored in data-files, record the filename, storage location (or repository), and version if applicable. **Important:** Raw measurement data should be preserved. Any processed or corrected data should remain traceable to the original measurements.
	processing	Process the raw data according to the procedure described in the Data Preparation section. Document each analysis

		step so that another person could reproduce the results.— include brief explanations, example calculations, or intermediate graphs where relevant. If you use a script (e.g. Python) to process your data include the name and location of the script, or if it is short, include the listing in your journal.
	error processing	Report the errors associated with every measured quantity and every reported result. Explain how each error was estimated (e.g., instrument precision, error propagation). Include at least one representative calculation demonstrating how the error of a derived quantity was obtained.
Reflection	**Discussion and Conclusion**	
	discussion	Interpret the results presented in the previous section. Discuss observed trends, relationships, and anomalies, and assess whether they are consistent with (theoretical or accepted literature) expectations. If discrepancies are observed, identify and explain possible causes (e.g., experimental errors, assumptions, limitations in the setup). **Important:** The discussion should interpret previously presented results only. Do not introduce new measurements, calculations, or observations.
	conclusion	Draw conclusions that directly address the goal of the experiment. . Give a summary of the key results. Report the final values of the determined quantities together with their uncertainties in numerical form or refer to a graph. Indicate to what extent the goal of the assignment has been reached.
	recommendations	Provide realistic and relevant recommendations to improve the measurement method. Justify each recommendation by referring to a limitation or source of uncertainty identified in the discussion. Base your suggestions on, for example, earlier identified error sources or other issues observed during the experiment. Note: Recommendations are realistic if they can be implemented within the scope of a student lab with the available resources (E.g. get a better, more expensive instrument is *not* realistic).

6.4 Journaliseren bij een ontwerp-opdracht

Ontwerpen is een proces waarbij een probleem (of uitdaging) voorop staat en waarvoor een oplossing gevonden moet worden. Vaak is hier sprake van een opdrachtgever die het probleem omschrijft met eventuele randvoorwaarden en restricties. De opdrachtnemer analyseert de vraag en bedenkt een oplossing. Deze oplossing wordt dan gebouwd, waarna het resultaat getest wordt en gevalideerd wordt tegen de vraag en gestelde eisen.

Een probleem moet vaak in meerdere deelproblemen opgesplitst worden. Voor deze deelproblemen worden deeloplossingen bedacht, die tesamen de eindoplossing vormen.

Tijdens onderzoek wordt ook ontworpen. Denk bijvoorbeeld aan de meetmethode. Hieraan worden eisen gesteld en de student moet een oplossing bedenken waarmee de metingen volgens de eisen uitgevoerd kunnen worden.

Het test- en evaluatieonderdeel van het ontwerpproces heeft de kenmerken van een onderzoek. De verwachting moet geverifieerd worden.

6.4.1 De standaard journaalstructuur bij een ontwerpopdracht

- titel	Vermeld het opdrachtnummer en de titel van de proef.
- samenwerking	Als er wordt samengewerkt, moet de naam van de ander vermeld worden.
- datum	Vermeld de datum en eventueel de tijd.
<u>Analyse fase 1:</u>	
- (ontwerp)doel	In het doel staat wat er precies ontworpen en gemaakt moet worden, zoals door de opdrachtgever is aangegeven
specificaties	Bij specificaties worden zowel de volgende zaken benoemd: wat zijn de eisen (wat moet het ontwerp doen), randvoorwaarden (onder welke omstandigheden moet het ontwerp functioneren), beperkingen (wat beperkt je in ontwerp vrijheid, denk aan budget, tijd, materiaal, ect....).
	LET OP: In deze stap wordt nog niet naar de analyse of de oplossing gekeken
<u>Analyse fase 2</u>	
- Doorgronden van het probleem	
	- benodigde theorie om de basisprincipe van het ontwerp te onderbouwen
	- uitdagingen die voortkomen uit de eerder gestelde randvoorwaarden
	- obstakels die voortvloeien uit de eerder gestelde beperkingen
Tijdens deze fase kunnen al wat korte testen gedaan worden om bijvoorbeeld werkingsprincipes of	haalbaarheid van een (sub)onderdeel te testen.-
- meetbare eiesen	Beschrijf voor ieder subonderdeel de hun doel en werking binnen het geheel. Beschrijf per subonderdeel kort hoe deze gebouwd en getest gaan worden.
<u>De synthese:</u>	
- bouwen	In de synthese begin je met het bouwen van je ontwerp. Hiervoor bouw je eerst een subonderdeel, deze test je vervolgens. Het kan zijn dat afhankelijk van een uitkomst

van een van je testen, bepaalde waarden opnieuw berekend moeten worden, dat doe je ook bij deze fase.

- (deel)testen — Voor ieder los subonderdeel, zoals beschreven in analysefase 2 , doorloop je een testfase. Dit kan soms redelijk kort en beknopt, bij wat meer ingewikkelde subonderdelen kost dit meer tijd. Tijdens de synthese schakel je dus de hele tijd tussen bouwen en testen. Iedere testfase bevat de volgende onderdelen:

  - doel — Geef hier een concreet **doel van de test** aan.
  - verwachting — Wat verwacht je voor concreet resultaat te meten.
  - theorie — De theorie horend bij de test die je gaat uitvoeren **(dus niet de theorie van je ontwerp!)**, dit kan dus ook een meetmodel en bewerking bevatten
  - meetprocedure — Dit kan vaak redelijk beknopt
  - meetresultaten — Hier beschrijf je dat wat je gemeten hebt, indien nodig geef je ook nog berekeningen en een bespreking, dit hangt af van je theorie en metingen.
  - conclusie — De conclusie **van de test**

<u>Test-fase prototype:</u>

- bouwbeschrijving — Tijdens de synthese ben je bezig geweest met het bouwen van een prototype, zorg dat je hier duidelijk en overzichtelijk je uiteindelijke prototype beschrijft. Iemand anders moet in staat zijn om, aan de hand van deze beschrijving, je prototype na te maken.

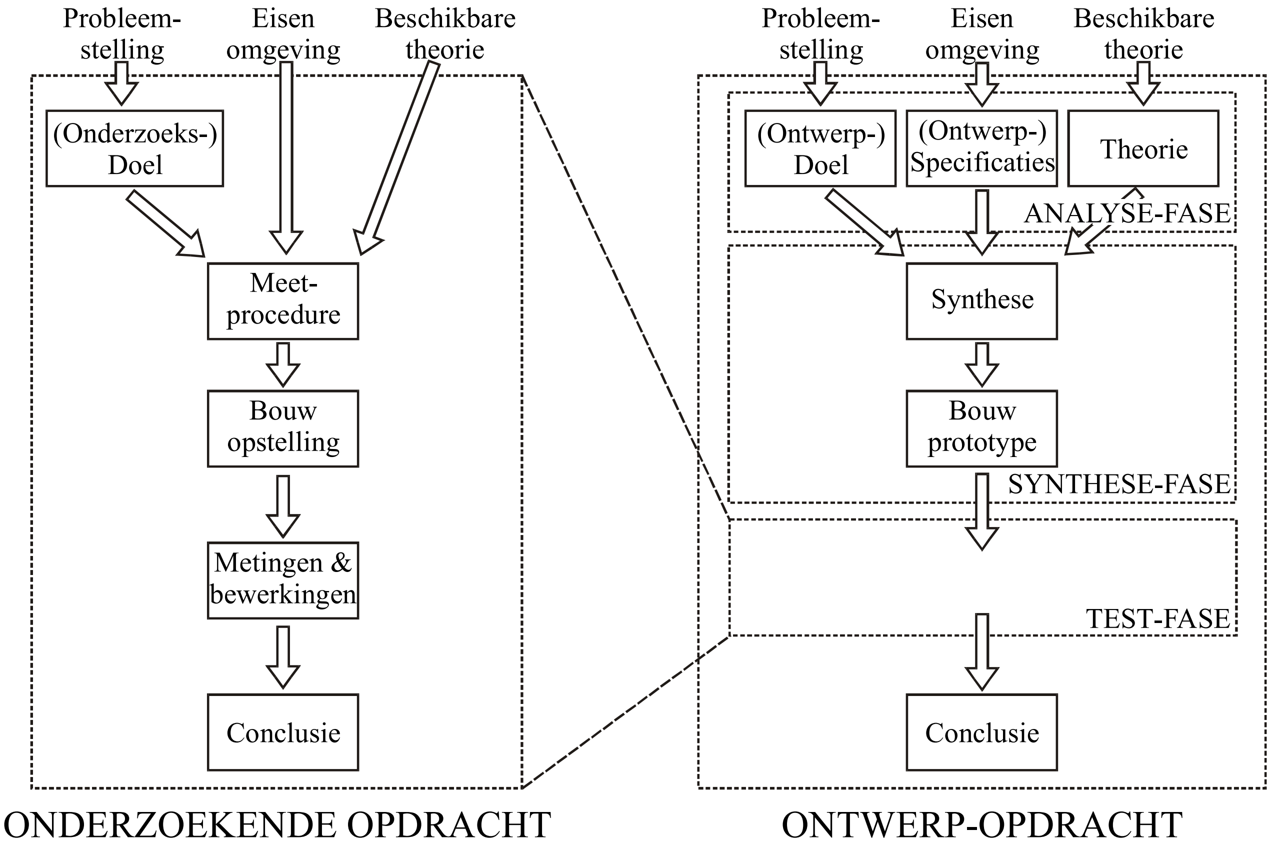
- testfase — In de testfase wordt het prototype getest. Om iets te kunnen testen moet er een experiment uitgevoerd worden waarbij aan een doel moet worden voldaan en er een bepaalde verwachting voor het resultaat is dat behaald moet worden. In principe volg je hiervoor de (versimpelde) journaalstructuur van een onderzoekende opdracht.

<u>De conclusie:</u>

- conclusie ontwerp — Vanuit de conclusie van de test kan je een **conclusie trekken over jouw ontwerp**. Hoe functioneert het ontwerp. Verwijs hierbij altijd naar de specificaties Geef ook aanbevelingen voor verbetering van het ontwerp.

6.4.2 *Grafisch verband tussen de twee journaliseermethoden*

In figuur 1 is een grafisch overzicht van de structuur van de behandelde twee journaliseermethoden gegeven. Duidelijk te zien is dat de test-fase bij een ontwerpende opdracht dezelfde structuur kan hebben als het journaal van een onderzoekende opdracht.



figuur 1 – Journaalstructuur van een onderzoekende en een ontwerp-opdracht

6.5 The standard journal structure

In this section two examples of journals are given, which follow the standard journal structure; one journal of a physics experiment and one of a chemistry experiment. In this examples can be seen that not all parts of the standard structure are necessary every time.

6.5.1 *Example journal for a physics experiment*

Preparation: *The assignment is: "Determine the electrical resistance of a (given) carbon resistor". Available are: a DC power supply and two multi meters. A reference is made to the laboratory manual for Ohm's law and the color code on resistors.*

Assignment 000: Resistance determination

Date: February 29, 2024 Partner: Xxxx Xxxxxxx

Goal: Determine the electrical resistance of a carbon resistor as accurately as possible by measuring the voltage and the current.

Expectation: The color code provides an estimated value that will be given with the experimental setup. This value has a maximum tolerance of 10% and will correspond to the determined value within the error range.

Measurement method

Measurement model:

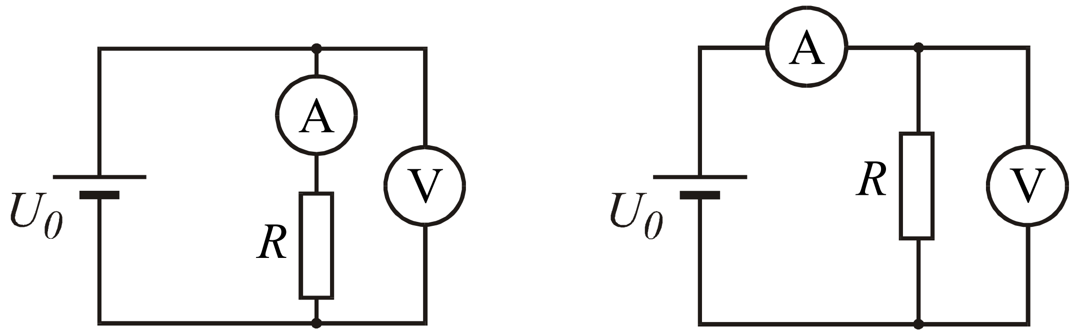


Figure 1 Schematic representation of Setup. Where A is the ampere meter to measure the current I and V indicate the voltmeter to measure the voltage U.

Example of a table. Taken from the Reader Data analysis (TN).

For determining the resistance Ohm's law can be used

$$U = IR \tag{1}$$

with U = voltage in volts (V)
I = current in amperes (A)
R = resistance in ohm (Ω)

One limiting factor for Ohm's law is that the temperature must not increase (significantly), otherwise the resistance will change.

From Ohm's law the model for the experiment follows: R=U/I

(This formula has the correct form if only one single U and I value is measured and R is calculated directly from them. It is better to plot a number of U and I measurements in a graph and determine R from the slope. The measurement model can then be skipped because in the 'theory' the formula has already the right form.)

To determine R, U and I have to be measured for different values of U.

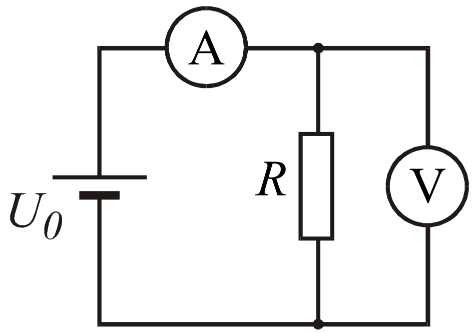
Data Preparation U and I are both measured for multiple datapoints and the errors will be recorded. U will be plotted on the y-axis and I on the x-axis as indicated in formula (1). From the slope R can be determined.

Error analysis:
For figure 1: suppose U=15V (maximum), then I=15V/470Ω ≈ 30 mA. According to the equipment documentation the voltage drop over the ampere meter in the range 0-30 mA is approximately 100 mV. That is about 1% of 15V, so about the class error.
For figure 2: in the range 0-30V, the voltage meter has an internal resistance of 3.16 MW. This about 6000×R. So, the ampere meter produces an error of 1/6000. This is much smaller than the previous circuit.
Conclusion: we choose the circuit from figure 2. The induced error is much smaller than the class errors of the meters. We don't have to correct for the systematic error.

ΔU and ΔI follow directly from the accuracies of the meters. These can be found in the manuals. ΔR follows from the *least square fit* given by the fitting program.

Experimental

Setup



U_0 – DC power supply Delta E015-2, range 0-15 V, 0-2A
I and U - multi meters Unigor-A43
R – Some carbon resistors. We chose a resistor of 470W ± 10% (color code: yellow-violet-black-black-silver)

Measurement procedure: The source voltage U_0 will be varied between 1-15 V with intervals of 2V. For every U0 the current I through the resistor and the voltage U across the resistor will be measured with a multi metar as shown in the figure of the setup.

Results

Mearuments and observations Measurements have been performed on a 470Ω resistor. For U≈15V, I≈30mA. The power developed in the form of heat is P=450 mW. A carbon resistor can handle up to 0.6 W, so this is no problem.

8 measurements have been done between 0 and 15 V in steps of 2 V. The results can be found in table 1.

The accuracy of the voltage and current measurement is specified in the equipment documentation. The accuracy is determined by the class error of 1% of the full scale. (the read off error is contained in the class error). For the 2V measurement in the 3V range : 1% of 3V gives an accuracy of 0.03 V. The other errors are calculated in a similar way, see table 1.

Table 1: Measuring the resistor R

U(V)	Range(V)	DU (V)	I(mA)	Range(mA)	DI (mA)	
1.00	1	0.01	2,05	3	0.03	
2.00	3	0.03	4,2	10	0.1	
4.00	10	0.1	8,5	10	0.1	
6.00	10	0.1	12,7	30	0.3	
8.00	10	0.1	18,4	30	0.3	Measured
8.00	10	0.1	16,4	30	0.3	again
10.00	10	0.1	21,0	30	0.3	
12.00	30	0.3	25,2	30	0.3	
14.00	30	0.3	29,3	30	0.3	

The results have been plotted in graph 1. (U,I)=(0,0) is also a point in the graph (check on instrument zero).

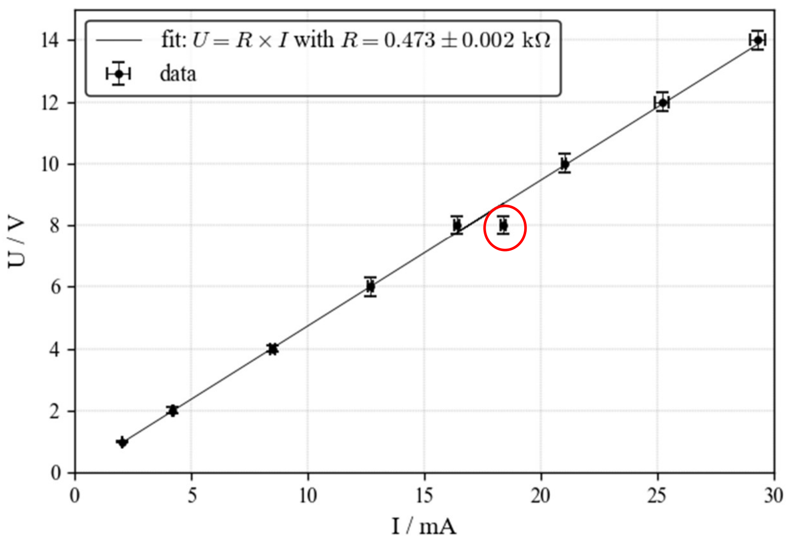


Figure 2. Relation between voltage across and current through a resistor to determine the resistance of the resistor.

The data point at U=8.00 V doesn't seem to be correct. Another measurement has been taken, giving a current of 16.4 mA. This result is consistent with the other data. There must have been a wrong reading of the meter.

Error processing

The errors in U and I are plotted with the datapoints in the graph and the resistance is determined with the slope. The fitting program calculated the least square fit between the data points and the slope. This gave $R = 473 \pm 2\ \Omega$

Discussion and Conclusion

Discussion The data follows the linear trend where it has the cut of at (0,0), as expected from the theory. The datapoint of U=8.00 V doesn't match the fit. It could be that the reading of the meter wasn't correct and therefore the datapoint was remeasured.

Conclusion The obtained value is $R = 473 \pm 2\ \Omega$. This result is consistent with the color code of the resistor.
The errors in the measurements can be completely attributed to the class error of the instruments.

Recommendations: The measurements are already accurate. Only more datapoints across the whole range and/or more datapoints per source voltage U_0 to minimize random errors.

6.5.2 *Example journal for a chemistry experiment.*

Assignment 1

Date: 7 march 2016 Partner: Xxxx Xxxxxxxx

Goal: Determination of the composition of a mixture of glutaric acid and malonic acid.

Measurement method

Theory

Malonic acid and glutaric acid are both dicarboxylic acids. The chemical formulas are as follows:

$HOOC\text{-}(CH_2)_3\text{-}COOH$	$HOOC\text{-}CH_2\text{-}COOH$
Glutaric acid	Malonic acid

The densities are known: glutaric acid = 1,424 g/cm3 and malonic acid = 1,619 g/cm3
The molar masses are known: glutaric acid M=132 g/mol, malonic acid M=104 g/mol

Measurement model

We need some quantity that depends on the composition and also can be measured. Possibilities are:

a) Density Determining the density of powders is experimentally a bit tricky, so not very suitable.

b) Melting point The melting temperature is not proportional to the composition. One would need to prepare a large range of mixtures with known composition. A relation between the melting temperature and composition could be determined experimentally and that could be used to determine the composition from the melting temperature of an unknown sample. This method is ok in principle, but a lot of work.

c) Average molar mass The average molar mass of a mixture of acids can be determined using acid-base titration. When only two acids are present and the molar mass of these is known, the exact composition can be determined.

Method c) will be used, since it is the easiest method to execute.

If the composition of the mixture of glutaric acid/malonic acid = x : y (mol%) then:

$$M_{mixture} = \frac{x}{100}132 + \frac{y}{100}104 \quad (1)$$

Moreover x + y = 100, thus

$$M_{mixturel} = \frac{x}{100}132 + \frac{100-x}{100}104 \quad (2)$$

or:

$$x = \frac{100}{28}(M_{mixture} - 104) \quad (3)$$

Measurement of $M_{mixture}$ leads to x.

In this case, acid-base titration is a suitable and relatively fast method.

If we use v ml NaOH (b mol.l^{-1}) for the titration of a gram mixture then:

Moles acid = moles NaOH (dibasic acids), thus:

$$\frac{a}{M_{mixture}} = \frac{1}{2}vb \cdot 10^{-3} \quad (4)$$

and so:

$$M_{mixture} = \frac{2a}{vb \cdot 10^{-3}} \quad (5)$$

Substituting in (3) gives:

$$x = \frac{100}{28}\left(\frac{2a}{vb \cdot 10^{-3}} - 104\right) \quad (6)$$

Measurement procedure

If a NaOH solution with known concentration (b) is available, only a (with a scale) and v (with a burette) have to be measured. As indicator phenolphthalein is used, because both acids are weak and the pH of the second equivalence point will be >8. If the measurements are done with a burette with max 10ml 0,1M NaOH then a maximum of ½·10·0,1000 mmol acid has to be weighed, i.e. about 50mg ($M_{mixture}$ is between 104 g/mol and 132 g/mol).

Measurement and operation:

Step by step directive

Weigh approximately 50mg of mixture on an analytical scale (4 decimal places), transfer this quantitatively into an Erlenmeyer flask of 100ml and add approximately 25ml of distilled water. Fill the burette with 0.1M NaOH (3x rinsing), add 3 drops of phenolphthalein and titrate until the color changes (light pink). Do this five times.

Safety

In the chemiekaartenboek the safety information of glutaric acid and malonic acid have been checked. The substances are irritating (glutaric acid) or corrosive (malonic acid) for eyes, skin and respiratory organs. Direct contact with the substances should therefore be avoided. If this should happen after all, it has to be rinsed off with water. Solutions of these substances are corrosive.

Solutions with these substances should be discarded in waste category I (acid and neutral inorganic substances in solution).

Error analysis

We use an internal error estimate on the basis of multiple measurements.
The standard deviation can be calculated which gives:

$$s = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(x_i - x)^2} \qquad (7)$$

For the standard deviation of the mean is given by:

$$s_x = \frac{s}{\sqrt{n}} \qquad (8)$$

Calculation of the 95% confidence interval goes according the rules of error analysis.

Experimental

In the titration a piston burette of 10 ml of class A / NEN 1755 is used.

Results and discussion

Table 1 contains the results of the measurements and calculations. The mole fraction glutaric acid , x, has been calculated using eq. (6).

Table 1 : Measurement results

Measurement	Mass (gram) Container full	Mass (gram) Container empty	a(mg)	v (ml)	$M_{mixture}$ (formula 5)	remark
1	7,1823	7,1354	46,9	7,44	126,1	
2	8,9261	8,8801	46,0	7,26	126,7	
3	7,0927	7,0396	53,1	8,81	120,5	Too far
4	8,7831	8,7271	56,0	8,80	127,2	
5	8,6432	8,5884	54,8	8,68	126,3	

The third measurement is not included because the measurement is gone beyond the equivalence point.

The average of the mole fraction glutaric acid is calculated on the basis of measurements 1,2, 4 and 5: x = 80.7 mole%

Error processing

From eq. (7) it follows for n=4, that the standard deviation is s = 1.92 mole%

From eq. (8) it follows that the standard deviation in the mean is s_x = 0.96 mole%.

The t-value for the 95% confidence interval follows from the table in the GLM. The number of degrees of freedom f=3, so, t = 3.182. From this it follows that the 95% ci = 3.2×0.96 mole% = 3.1 mole%.

Rounded, the mole fractions are therefore: 80 ± 3 mole % glutaric acid and 19 ± 3 mole% malonic acid.

Conversion to mass fractions

100 mol mixture contains 81 ± 3 mole glutaric acid and 19 ± 3 mole malonic acid. Or, 81 ± 3 × 132 gram glutaric acid and 19±3 × 104 gram malonic acid. This is 10692 ± 396 g glutaric acid and 1976 ± 312 g malonic acid. Dividing by the total mass of 12668 g gives the following mass fractions:

84±3 % m/m glutaric acid and 15±3 % m/m malonic acid.

Conclusion

The composition of the unknown mixture of glutaric acid and malonic acid has been determined using acid-base titration. The mole fraction were 81±3 mole glutaric acid and 19±3 mole malonic acid (95% ci).

This corresponds to 84±3 % m/m glutaric acid and 15±3 % m/m malonic acid.

The error can be reduced by doing more measurements or by using larger quantities of acid (Schellenbach burette 50ml).

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