

AALTO UNIVERSITY
School of Chemical Technology
Department of Chemistry
Physical Chemistry Laboratory

GENERAL INSTRUCTIONS FOR WRITING THE LABORATORY REPORTS

Aalto University
 School of Chemical Technology
 Department of Chemistry
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ATTACHMENTS

Attachment 1. Laboratory notebook

1 Structure of the Report

Name Page

The first page of the report is the name page which is filled appropriately. The name pages can be downloaded from the webpages of the course or they can be collected from the laboratory.

Introduction

The background of the lab work, the work itself as well as the purpose of the lab work are briefly introduced in the introduction (max. 1 page). This section does not include any equations.

Theory

The most appropriate equations -and especially the ones that are used later in the report- are shown in this section. There must also be short explanations of the equations.

The important equations with respect to the experiments and results are derived from generally known theory. More detailed derivations of the equations, which are shown in work instructions or in books, are not demanded. It is enough to give the appropriate equations with references.

Experimental Equipment and Methods

The equipment, experimental methods and materials used are introduced in this section.

Results

All the results from the laboratory notebook are collected and shown, for example in the form of a table.

All the calculations – including the intermediate steps – which are needed to reach the final result are shown. Additionally, an example calculation with proper values and units must be shown for every equation.

When using an equation, the Theory section must be referred to or the origin of the equation has to be shown by other means.

All the results (and possible intermediate results) are shown, for example in tables and/or figures.

Error Estimation

An estimation of the reliability of the results is added to all the results and the biggest sources of errors must be clearly explained.

Discussion and Conclusions

The most important results with their error limits are shown in this section. Also, the results are compared to the literature values if available. All the literature values must be referenced in the usual way.

Results are discussed: Are the results in good accordance with literature values? If not, what is the reason?

Answers to the Questions

The answers to the homework questions are shown in this section.

References

The references are listed in the order of appearance in the script or in alphabetical order. All the listed references must be mentioned in the report text.

Attachments

All the additional pages are added as attachments: the page of original laboratory notebook, printed data sheets from the computer, graphs from the graphic plotter, etc.

Corrections

All the corrections for the report are made on separate pages and attached to the back of the original report by stapling.

2 The Format of the Report

2.1 General

The report is printed single sided on A4 paper. Text is written in a single column with line spacing 1.5 and the text is justified. The left margin is 3.5cm and all the other margins are 3 cm. Page numbers are placed in the centre either at the top or bottom of the page. The name of the lab work is written in font size 16, bold, using capital letters and it is centred. Main headings are written in font size 14 and bold, the subheadings are written in font size 12 and bold, the text itself is font size 12. The fonts of figure and table captions are identical to the body text. Before all the headings, there are two empty lines. Between the paragraphs there is an empty line and text starts from the beginning of the row.

The readability of the text is important. Text must be short, concise and grammatically correct. All the sentences must be whole sentences. The passive and imperfect tenses are always used in the text.

SI units are used whenever possible. It is not permissible to use the format 5E-6m, neither in the text nor in tables, the formats 5×10^{-6} m or 5 mm should be used.

Decimal points are used in numbers and they are rounded to reasonable levels, depending on the errors in the measurements.

2.2 Units and Equations

Equations and reactions are numbered by continuous numbering and a number is placed in parentheses on the right side of the page. Equations are written by equation editor (i.e. using appropriate symbols, not letters).

Equations must be explained in the text prior to inclusion. The number of the equation is separated in text using the parenthesis, i.e. Equation (1).

Equations can be shown either as equations between quantities or dimensionless equations with quantities. Dimensionless equations with quantities must be used always when the equation is not homogenic with dimensions, i.e. the units are not cancelling themselves out, as shown for example in Equation (2).

An example of the equation between quantities:

$$q_P = n C_{P,m} \Delta T \quad (1)$$

where q_P heat transferred

n molarity

$C_{P,m}$ molar heat capacity

ΔT temperature difference

An example of the dimensionless equation with quantities:

$$\frac{C_{P,m}(\text{H}_2, \text{g})}{\text{J K}^{-1} \text{mol}^{-1}} = 29,064 - 0,8363 \times 10^{-3} \frac{T}{\text{K}} + 20,111 \times 10^{-7} \frac{T^2}{\text{K}^2} \quad (2)$$

where $C_{P,m}(\text{H}_2, \text{g})$ the molar heat capacity of hydrogen gas at constant pressure

T temperature

An example calculation must be shown for each equation first with measured or other values and dimensions. The workings of the calculation have to be shown in such a way that the final value with the units can be checked and corrected when necessary. When using a literature value, a reference must be mentioned.

An example calculation:

Equation (1) gives

$$q_p = 75,3 \frac{\text{kJ}}{\text{K mol}} \times 555 \text{ mol} \times (323 - 283) \text{ K} = 1670 \text{ kJ}$$

2.3 Figures and Tables

There is a separate numbering for figures and tables throughout the text.

All the figures and tables must be referred to in the text before showing them for the first time.

Figure and table captions have to be understood without the report text. The font of the figure or table caption is exactly the same as the body text. **The figure caption is placed under the figure and the table caption is placed above the table.**

The title and units for every row and column must be mentioned in the tables. The rows must be vertically aligned with respect to a decimal point and too many digits should be avoided.

An example of a figure:

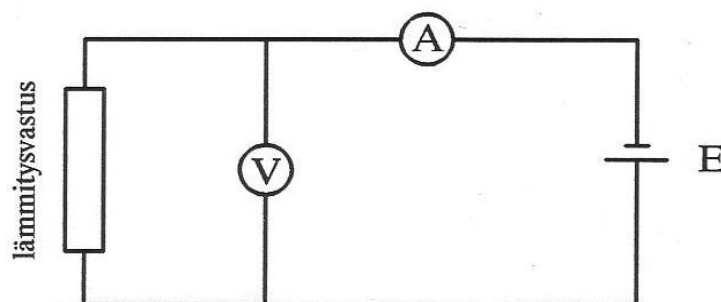


Figure 1. Coupling of calorimeter for the determination of heat capacity.

V is a voltmeter, A an ammeter and E the voltage source.

An example of a table (according to IUPAC [1] recommendations):

Table 1. The dependence of n-propanol vapour pressure P on temperature T .

Experiment	T / K	$10^3 \text{ K} / T$	P / kPa	$\ln (P / \text{kPa})$
1	333,15	3,0017	19,6	2,98
2	343,15	2,9142	31,3	3,44
3	353,15	2,8317	50,1	3,91

2.4 Graphs

Figure numbers must be shown for all the graphs and **the figure caption is placed under the graph.**

Measured or calculated data points (x_i, y_i) are illustrated graphically as shown in Figure 2. The error limits Δx_i and/or Δy_i are added to the data points by horizontal and/or vertical lines for every data point (x_i, y_i) if needed.

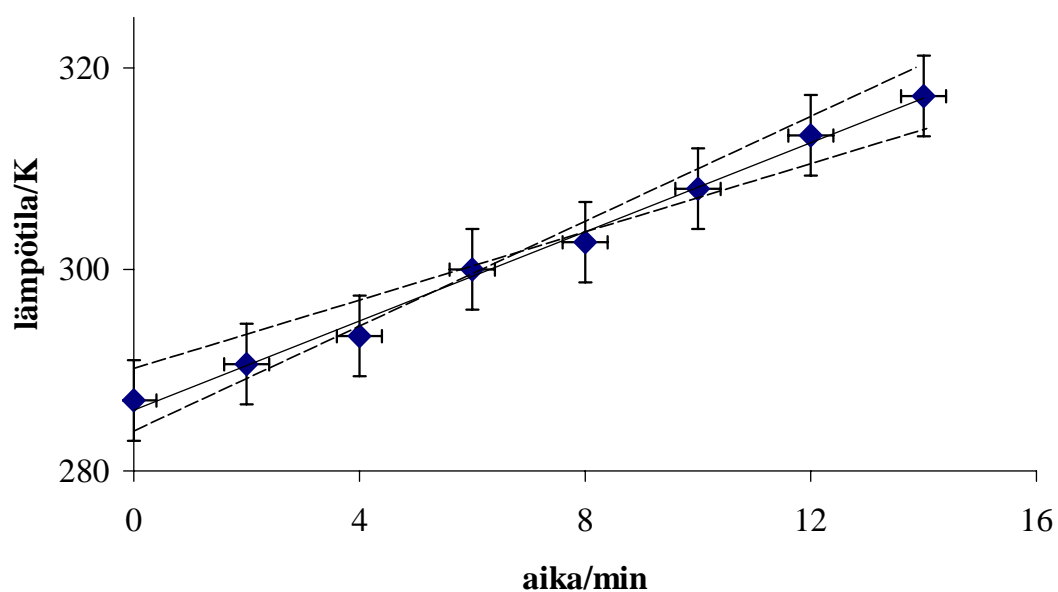


Figure 2. The increase of temperature as a function of time in experiment XX.

The axes and the scale of the graph are chosen appropriately. An appropriate scale is such that the graph fills the paper as widely as possible and for example, in the case of a straight line, the plot is approximately at angle of 45° when compared to the X axis. **No background colour or a box around the figure is allowed.**

Axes are labelled and the units are also shown.

When adding data points to the graph, each data point must be presented clearly. All the data points must be clearly visible after drawing the graph. If there are several data points at different conditions, one must use clear markers which are easily distinguishable from each other. Good markers are for example circles, triangles, squares, etc., as shown in Figure 3.

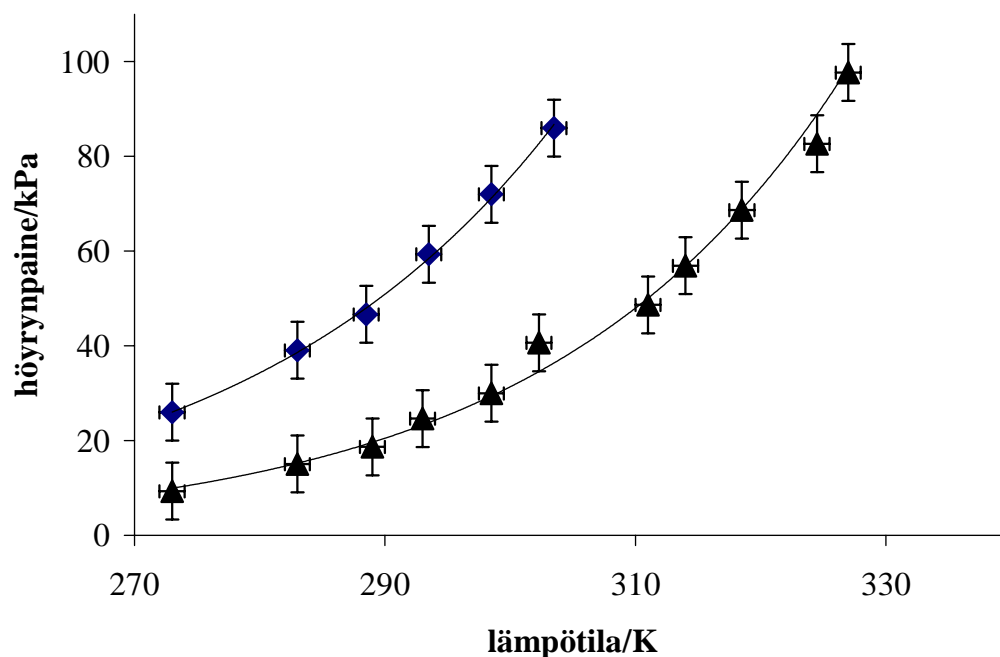


Figure 3. The dependence of the vapour pressure of a pure substance with temperature.

“ diethyl ether ▲ acetone

The curve which illustrates the best relation between the values is shown in graphs. When fitting the curve, obvious error points can be excluded from the fitting but these data points must be still clearly seen in the graph. As measured results always contain some error, data points rarely sit exactly on the fitted curve. **Thus, data points are not allowed to be connected to each other with a fraction line.**

If data points are fitted to give a straight line, it is a good idea to draw error lines into the graph (broken lines in Figure 2). Error lines cross each other at the focal point of the set of data points. If the measurements are performed at regular intervals and the error limits are approximately the same, the focal point is more or less in the centre of all the data points. It is also important to remember that the error lines should be within the error limits of each of the data points as often as possible. Error lines can also be drawn on the figure by hand.

2.5 Attachments

Attachments are added to the end of the report. Page numbers do not continue to the attachments but every attachment has its own number and title. Attachment number is written on the top right corner using capital letters (ATTACHMENT 1, ATTACHMENT 2, etc.). If one attachment contains several pages, it can be shown like the following: ATTACHMENT 1 (1/3). The note in parenthesis indicates the first page of three pages.

All the attachments must be referred to in the report text.

The figures and tables shown in the attachments are formatted according to Chapters 2.3 and 2.4. Each attachment is an independent entity, i.e. the numbering starts from 1 in each attachment.

Attachment 1 shows an example of the laboratory notebook. Laboratory notes can be made by hand using a ballpoint pen.

3 Data Handling and Error Estimations

The purpose of the laboratory work performed in the physical chemistry practicals is usually to find out the value of a certain physical property by experimental methods. Therefore, the results always contain some error. The size of the error and the main sources of the error are found by error estimation.

When estimating the errors, the reasoning behind the estimation must always be explained. It can be, for example, the precision of the equipment or the maximum error based on the total differential.

In this section a brief summary of the principles and methods of error estimation is outlined. More information can be found for example from the books of Vaari [2] (in Finnish) or Shoemaker *et al.* [3].

3.1 Error Types

The errors appearing in the experimental measurements are usually divided into rough, systematic and random errors.

Rough error is due to the inappropriate use of equipment, error in reading, etc. It is assumed to vanish when measurements are done carefully.

Systematic error always distorts the results in the same direction. Systematic error occurs, for example, when calibration of the equipment is not done correctly (so called calibration error) or if analogue scale is read from the wrong angle (so called parallax error). Systematic errors should try to be detected and corrected whenever possible.

Random error i.e. statistical error does not contort the result in one direction. Random error is in every experimental measurement and the existence can be

detected only by repeating the measurement several times. Estimation of random errors is done using statistical tools.

If the amount of systematic errors is small, the **trueness** of the measurement is said to be good. If the amount of random errors is small in the final result, the **precision** of the result is said to be good. In this case, the average of the repeated measurements should be close to the absolute value. The effects of systematic and random errors are illustrated graphically in Figures 4 and 5.

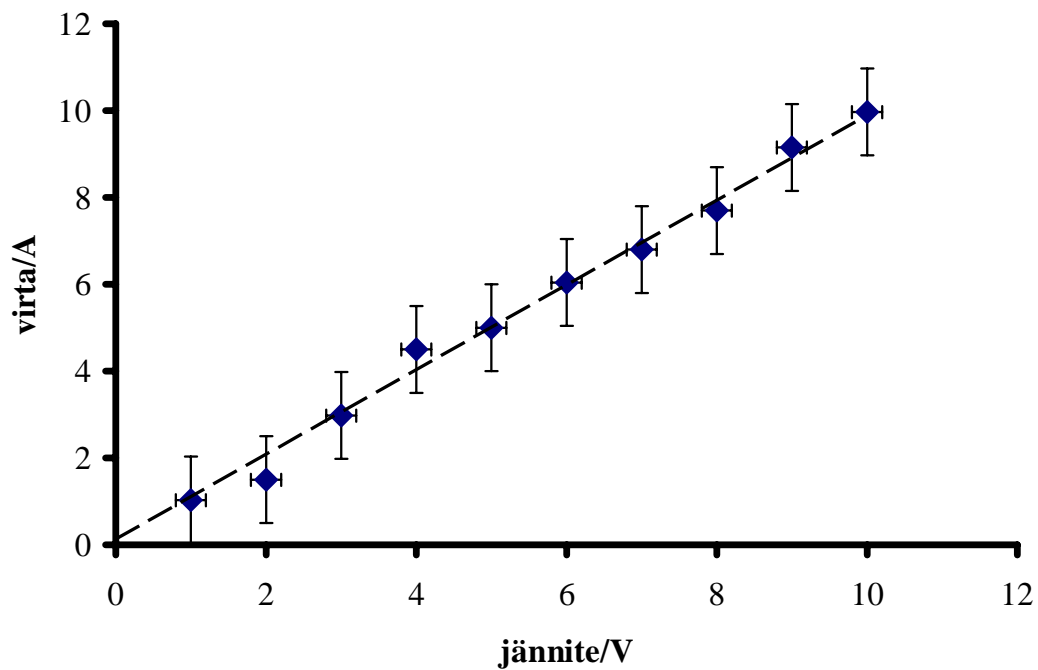


Figure 4. The systematic error is small as the theoretical line (broken line) illustrates the data points well, but the random error is big, as the error limits of each data point are large and there is clear scattering of data points.

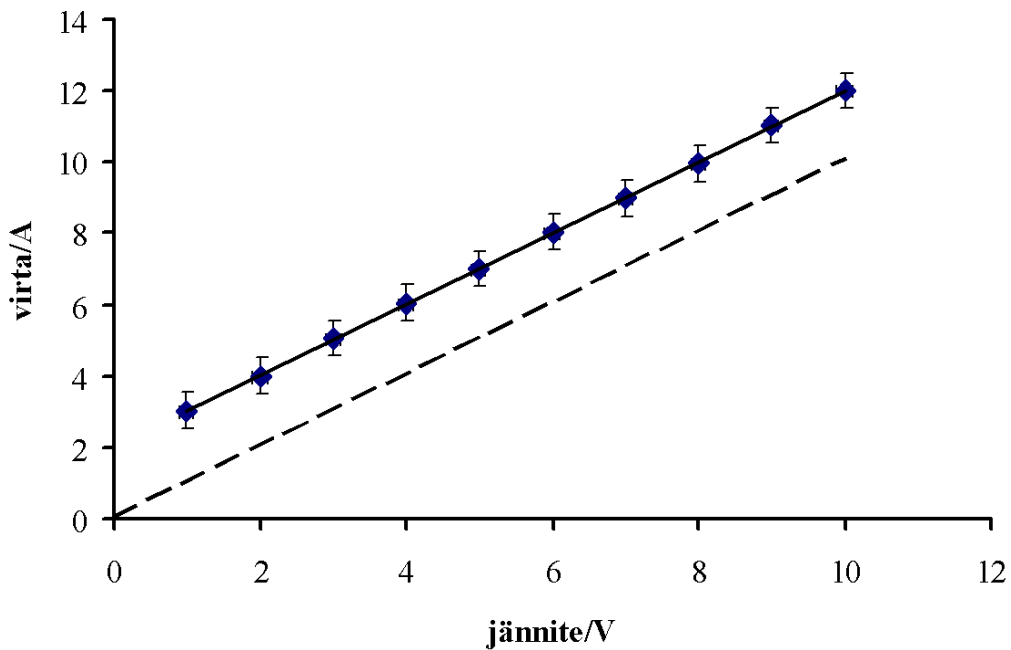


Figure 5. The random error is small because the error limits of data points are small and the data fits well on the same straight line. Systematic error is large, as the data does not fit on the theoretical line (broken line).

3.2 Estimation of the Error of Measured Values

The maximum random error for each measured quantity is estimated qualitatively (precision of the equipment, fluctuation of the value measured by the equipment, etc). Note that the error of the measurement is normally constant over the entire measurement range. Hence the error in the measurement is absolute and not relative. Here ‘the maximum of the random error for measured values’ means the absolute range within which the absolute value of the random error is at certain probability (for example, 95% or 90%). This value is an absolute part of the measurement range, for example 0.05 bar, and the measured value can be presented in the format $P=(1.53\pm0.05)$ bar. The error of a pressure is therefore given in bars and not as a percentage.

3.3 Error Estimation for Calculated Quantities

The quantities to be studied cannot always be measured directly. In this case the measurements are performed using experimentally accessible quantities which can deliver the desired quantity (by a certain function). The mathematical model which describes the phenomenon is assumed to be known and the studied quantity is calculated using a model and measured values.

3.3.1 Calculating Error Estimations using the Total Differential

It is assumed that the quantities $x_1, x_2, x_3 \dots$ are measured and the error estimates $Dx_1, Dx_2, Dx_3 \dots$ have been defined for these measured quantities. If there is a mathematical model describing the relationship between the quantity being studied and experimentally accessible quantities, $F = F(x_1, x_2, x_3 \dots)$, then the change in the function F with respect to the small change in the parameter x_i is defined by the derivative of F with respect to x_i . If there is a small change (dx_i) in all of the measured quantities, the change in the function F can be calculated using the total differential:

$$dF = \frac{\partial F}{\partial x_1} dx_1 + \frac{\partial F}{\partial x_2} dx_2 + \frac{\partial F}{\partial x_3} dx_3 + \dots \quad (3)$$

Partial differentiation is utilised in Equation (3), i.e. the derivative of the function is performed for each parameter separately while keeping the other parameters constant.

When calculating the final error for function F , the differentials in Equation (3) are replaced by the error limits (Dx_i). The direction of the error is unknown but a maximum limit can be found when all the errors are kept positive. Additionally, as the purpose is to calculate the maximum of the error, all partial derivatives are absolute

values. If this was not done, opposite terms might cancel each other out. Error estimation for the calculated value can be then calculated by the equation:

$$DF = \left| \frac{\partial F}{\partial x_1} \right| Dx_1 + \left| \frac{\partial F}{\partial x_2} \right| Dx_2 + \left| \frac{\partial F}{\partial x_3} \right| Dx_3 + \dots \quad (4)$$

For example:

Calculate the error estimation for the function

$$F = \frac{xy}{x+y}$$

$$DF = \left| \frac{\partial F}{\partial x} \right| Dx + \left| \frac{\partial F}{\partial y} \right| Dy = \left| \frac{y^2}{(x+y)^2} \right| Dx + \left| \frac{x^2}{(x+y)^2} \right| Dy \quad (5)$$

3.3.2 Calculation of Error Estimation by Logarithmic Derivative

If the mathematical model contains only multiplication, division and involution, the error estimation can be done by determining the logarithmic derivative. It usually decreases the number of calculations in error estimation. Mathematically using the logarithmic derivative and total derivative yields identical results and therefore, both give exactly the same value for the error estimation.

With the logarithmic derivative, the logarithm of the function is taken first and then the derivative.

For example:

Calculate the error estimation for the function $F = ka^a y^b$ where k , a , b and c are positive or negative real numbers.

First, take the logarithm of both sides:

$$\ln F = \ln(kx^a y^b) = \ln k + a \ln x + b \ln y$$

Then the derivative of both sides of the function are determined and the terms are placed inside absolute signs:

$$\frac{DF}{F} = \left| a \frac{Dx}{x} \right| + \left| b \frac{Dy}{y} \right| \quad (6)$$

Expression 6 gives the relative error DF/F with respect to relative errors of x and y . The absolute error DF is found by multiplying the relative error with the final result.

3.4 Studying the Dependence between the Quantities

The best and most commonly used method to find out if the results follow the behaviour predicted by the model, is to interpret the data graphically. With graphic illustration it is often easy to study the range in which the model is functioning, detect systematic and rough errors, calculate the values of model parameters and estimate their errors.

If possible, it is desirable to try to formulate the dependence between the quantities in the form of a straight line so that the desired quantity is in the slope. The slope and the constant term of the straight line can be determined by fitting a line to the set of data points either graphically or using the least squares method. From a graphical illustration which is drawn in the format of straight line it is also relatively easy to see if the results correspond with the theoretical model.

If, for example, the dependency $y = ax^2$ is studied and the constant a is to be determined, quantity y is drawn as a function of x^2 . In this case the data points should lie on a straight line in (x^2, y) co-ordinates. The slope of the straight line is then the desired quantity a . Error estimation (Dx and Dy) is performed according to chapter

3.2 and the error limit for the function x^2 is obtained by the method explained in Chapter 3.3, $D(x^2) = 2xDx$.

3.4.1 Determining the Slope and Intersect and Estimating Their Errors from a Graphical Illustration

Measured or calculated data points (x_i, y_i) are plotted on a graph as shown in Figure 6. The error limits Dx_i and/or Dy_i are added to data points by horizontal or vertical lines.

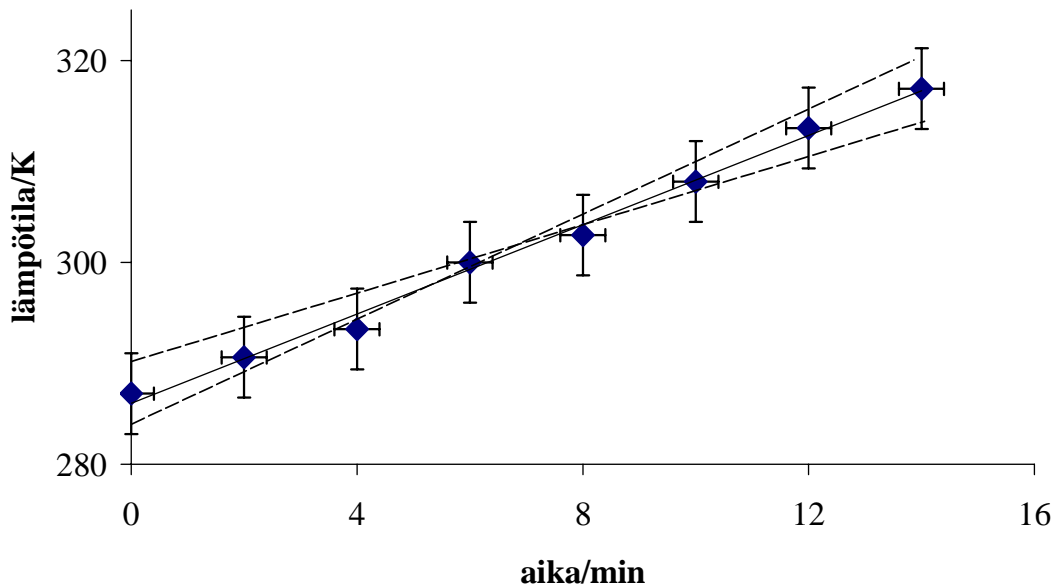


Figure 6. Illustration of how to determine the slope, intercept and their error estimations from a graphical illustration.

When the straight line which fits the data points optimally is drawn, the slope k can be determined graphically. For the determination of the slope two points which are relatively far away from each other and are not measured data points are selected.

The value of the slope is usually determined by inserting the values and units of the selected data points into the equation:

$$k = \frac{y_2 - y_1}{x_2 - x_1} \quad (7)$$

When error lines (broken lines) are drawn into the graph according to the instructions in page 10, the error of the slope can be calculated. The maximum k_{\max} and minimum k_{\min} of the slope can be obtained from the slopes of the error lines. The error of the slope Δk can be estimated as a maximum error:

$$\Delta k = \left| \frac{k_{\max} - k_{\min}}{2} \right| \quad (8)$$

Occasionally it is valuable to determine the value of the constant b for a straight line $y = kx + b$. The constant b can be obtained from the intercept of the y-axis and the straight line. The error limits can be determined by the intercepts of the error lines and y-axis. These error limits can easily become rather large, especially when the measurements are done far away from the y-axis. If the constant term must be determined from the straight line, it is good to ensure that the measurements are done close enough to the origin.

As a result, the slope and intercept can be obtained with their error limits, $k \pm \Delta k$ and $b \pm \Delta b$. If the data points lie on a straight line in the appropriate co-ordinates, this shows that the tested model is valid over the measurement conditions.

3.4.2 Least Squares Method (LSM)

To fit a straight line (or generally, any kind of function) to a set of data points there are several different types of algorithms. One of the most commonly used algorithms

is the Least Squares Method (LSM) which can be found in several computer programs and calculators (for example, Excel uses LSM).

LSM is a purely mathematical algorithm, which works exactly in the same way whether the graph has been drawn or not. Using LSM a straight line can be fitted to any kind of set of data points! Graphical illustration is necessary, to observe systematic and rough errors as well as to find out if the measured datapoints are in the range in which the theory is valid. If fitting a function to measured datapoints one must be sure that the model really describes the data points. This can be ensured **first** by making a graphical illustration.

A curve $y = F(x, a_1, a_2, a_3, \dots)$ is fitted into the set of data points (x_i, y_i) ; $a_1, a_2, a_3 \dots$ are the parameters of the curve. At the point x_i the residual (i.e. the vertical difference between the curve and data point) is $y_i - F(x, a_1, a_2, a_3 \dots)$. Using LSM, the best fit is found by minimising the sum S of the squares of all the residuals:

$$S = \sum_{i=1}^N (y_i - F(x, a_1, a_2, a_3, \dots))^2 \quad (9)$$

The minimum of the function S is found from the zero points of the derivative so that the constants $a_1, a_2, a_3 \dots$ can be obtained from the equations:

$$\frac{\partial S}{\partial a_1} = 0 \quad \frac{\partial S}{\partial a_2} = 0 \quad \frac{\partial S}{\partial a_3} = 0 \quad \dots \quad (10)$$

The most simple case for LSM is fitting a straight line into a set of data points in which there are no error limits for the points or they are all equal. In this case the fitted function is $F(x, k, b) = kx + b$ and the sum S to be minimised is

$$S = \sum_{i=1}^N (y_i - (kx_i + b))^2 = \sum_{i=1}^N (y_i - kx_i - b)^2 \quad (11)$$

Using Equation (10), the derivative of the sum S of the squares with respect to parameters k and b is determined and then the parameters k and b are solved from two equations. The following is then obtained:

$$k = \frac{1}{D} \frac{\sum_{i=1}^N x_i y_i - \frac{1}{N} \sum_{i=1}^N x_i \sum_{i=1}^N y_i}{\sum_{i=1}^N x_i^2 - \frac{1}{N} \left(\sum_{i=1}^N x_i \right)^2} \quad (12)$$

$$b = \frac{1}{D} \frac{\sum_{i=1}^N y_i^2 - \frac{1}{N} \left(\sum_{i=1}^N y_i \right)^2}{\sum_{i=1}^N x_i^2 - \frac{1}{N} \left(\sum_{i=1}^N x_i \right)^2} \quad (13)$$

where the parameter D is obtained from the expression

$$D = N \sum_{i=1}^N x_i^2 - \frac{1}{N} \left(\sum_{i=1}^N x_i \right)^2 \quad (14)$$

The statistically calculated values of the standard deviations Dk and Db for parameters k and b can then be found:

$$Dk = \sqrt{N \frac{s^2}{D}} \quad (15)$$

$$Db = \sqrt{\frac{s^2}{D} \sum_{i=1}^N x_i^2} \quad (16)$$

where s^2 is calculated as shown below

$$s^2 = \frac{1}{N-2} \sum_{i=1}^N (y_i - kx_i - b)^2 \quad (17)$$

LSM fitting gives the slope and the intercept of the straight line, $k \pm Dk$ and $b \pm Db$, using statistical mathematics.

4 Making References

Every fact, which cannot be assumed to be general knowledge for a chemical engineer, must have a reference. In the Physical Chemistry laboratory works a numbered reference list is favoured

A reference is inserted into the text which refers to the original source. The reference is marked using a number of the reference in square brackets. It may be worth adding the name of the author in the text before the reference number. If there many authors, the notation "first author's name *et al.*" is used.

Example of references in the text:

According to Christian [1], the concentration of hydrogen ions is calculated as the follows.... Corsaro [2] measured the velocity factor...

In the reference list, the references are presented as a numbered list and in the order of appearance in the text. A complete list of authors must be given.

An example of the reference list:

1. Christian, G.D., *Analytical Chemistry*, 4th Ed., John Wiley & Sons, New York 1986, p. 139.
2. Corsaro, G., A Colorimetric Chemical Kinetics Experiment, *J. Chem. Ed.* **48** (1964) 48-50.

5 References

1. IUPAC, *Quantities, Units and Symbols in Physical Chemistry*, editor I. Mills, T. Cvitas, K. Homann, N. Kallay and K. Kuchitsu, Blackwell Scientific Publications, Oxford 1988, p.3.
2. Vaari, J, *Fysiikan laboratoriotyöt, Suomen Fyysikkoseuran julkaisuja 4*, 3. edition, SFS Fysiikan Kustannus Oy, Jyväskylä 1998, pp. 2-30.
3. Shoemaker, D.P., Garland, C.W. and Nibler, J.W., *Experiments in Physical Chemistry*, 5th Ed., McGraw-Hill, Singapore 1989, pp. 29-82.

LABORATORY NOTEBOOK

LAB WORK 50. DETERMINING THE TRANSFERENCE NUMBER

Date: 22.9.2016

Lab group: Ernest Engineer and Sally Student

Author: Ernest Engineer

Table 1. Measured values

V / ml	t / s
0,03	178
0,06	341
0,12	690
0,15	856
0,18	1022
0,21	1201
0,24	1369
0,27	1535
0,30	1704