INTRODUCTION TO CHEMISTRY

LAB MANUAL

INTRODUCTION

The International System of Units (Systeme Internationale d'Unites); interconversion of SI and metric units

Metric units, which are often not identical to the latest international standards, are widely used among chemists and physicists around the world to record the results of their measurements. The basic metric units of length, mass, and volume are meter, gram and liter. When smaller or larger units are needed a system based on the powers of ten is used to form prefixes. The SI system of units is a modern and internationally accepted version of the metric system. In the SI system, the relationship between the different fundamental units is rigorously consistent. The main advantage of the SI system is that its exclusive use during calculations guarantees that any intermediate or final results for both fundamental and derived quantities will also be obtained in SI units without the need for conversion factors.

The following table gives the metric and SI units of the most important physical quantities.

Physical property	SI unit	Metric unit	Conversion
Length	meter (m)	meter (m)	
Volume	cubic meter (m^3)	liter (l)	$1\ l = 10^{-3}\ m^3 = 1\ dm^3$
Mass	kilogram (kg)	gram (g)	$1\ kg = 10^3\ g$
Pressure	pascal (Pa)	atmosphere (atm)	$1\ Pa = 1\ N/m^2$ 1 atm = 101325 Pa
		torr (mmHg)	$1\ torr = 1.333 \times 10^2\ Pa$
		bar	$1\ bar = 10^5\ Pa$
Temperature	kelvin (K)	Celsius degree (°C)	K = 273.15 + °C
Energy	joule (J)	calorie (cal)	1 cal = 4.184 J

Significant Figures

In science, it is fundamentally important to indicate the accuracy of measured or calculated data. The primary way of indication is to control the number of significant figures of a quantity. To do this, it is convenient to give all numbers in the common scientific notation using a factor between 1 and 10 multiplied by the appropriate power of 10. The number of digits in the first factor is the number of significant figures. For example:

$40200 = 4.02 \times 10^4$: the number of significant figures is three

The number of significant figures in any given quantity can be determined as follows:

(a) If there is no decimal point in the number, a count of the digits from the first non-zero digit on the right to the last non-zero digit on the left gives the number of significant figures.

(b) If the number contains a decimal point, a count of the digits from the first non-zero digit on the left to the very last digit (regardless of its value) on the right gives the number of significant figures.

Examples:

	Significant figures		Significant figures
5270	3	0.320	3
5027	4	0.32	2
0.0129	3	10.01	4

There are simple rules for giving significant figures in values calculated by mathematical operations. For multiplication or division the number of significant figures in the result is the same as the number in the least precise measurement used in the calculation. For addition or subtraction the result has the same number of decimal places as the least precise measurement in the calculation.

Accuracy and precision

Scientific measurements almost always have some error. This error may result from the limitation of the instruments used or the limitations of human senses. It is exceptionally rare to find that an experimentally measured value is exactly the same as theoretical predictions. The term ***accuracy*** is used to refer to the closeness of single measurement to the true value. The smaller the difference between the experimental and theoretical value the more accurate the results and the measuring device are. Some devices are more accurate than others. For example, 10 cm^3 of a liquid can be both measured by a single-volume pipette or a measuring cylinder. Pipettes are **more accurate** in this comparison.

However, the true value is only known if some kind of standard can be used for comparison. For most measurements, the true or theoretically predicted value is not known. This is why before the first use of any measuring device calibration must be done. This calibration is always in comparison with some kind of standard and makes sure that the new device is accurate enough. After careful calibration, the device can be trusted in further measurements.

Errors in measurements are almost always unavoidable. The best option for scientists to decrease the error of results is to do the same measurement several times (called parallel measurements) and calculate the average. The average is always more reliable than the result of a single measurement. For really reliable measurements, the parallel results are close to each other. ***Precision*** is used to refer to the closeness of the set of values obtained from identical measurements of a quantity on the same instrument. Precision is often given numerically as ***mean deviation***. To obtain mean deviation, first the mean value has to be calculated ($\bar{x}$), the absolute values of the individual deviations must be summed ($\sum\left|x_i - \bar{x}\right| = \sum\delta_i$ 0), and finally divided by the number of identical measurements ($\bar{\delta} = \frac{\sum\delta_i}{n}$).

For example:

During the calibration of a 5 cm^3 pipette, the following results were obtained: 5.041 cm^3, 5.033 cm^3, 5.019 cm^3, 5.021 cm^3, and 5.025 cm^3. What is the value of the mean deviation ($\bar{\delta}$)?

	measured values	deviation: $\lvert x_i - \bar{x} \rvert$
x_1	5.041	0.013
x_2	5.033	0.005
x_3	5.019	0.009
x_4	5.021	0.007
x_5	5.025	0.003
	mean: $\bar{x} = 5.028$	**mean deviation:** $\bar{\delta} = 0.007$

The calibrated volume of the pipette is given as: 5.028 ± 0.007 cm^3 or 5.028(7) cm^3 .

Interpolation, extrapolation

When two variables are connected linearly (f(x) = a·x + b), the value of the dependent variable (f(x)) can be calculated for any value of the independent variable (x) within the linear range provided that two pairs of independent and dependent values (x_1, f(x_1); x_2, f(x_2)) are known. The mathematical procedure is called **interpolation** if value x is within the known range (x_1 and x_2), and **extrapolation** if x is outside the known range.

A simple way to carry out the calculation is to note that $\frac{x_1 - x_2}{f(x_1) - f(x_2)} =$ constant for any pairs of variables. From the known x_1, f(x_1), x_2, f(x_2) values f(x) can be determined for any x value using the either of the equations $\frac{x_1 - x_2}{f(x_1) - f(x_2)} = \frac{x - x_1}{f(x) - f(x_1)}$ or $\frac{x_1 - x_2}{f(x_1) - f(x_2)} = \frac{x - x_2}{f(x) - f(x_2)}$.

Example:

Pl. The density of water for 20.3 °C is needed for a certain calculation. From standard tables it is known that the density of water at 20 °C: 0.99823 g/cm^3

the density of water at 21 °C: 0.99780 g/cm^3

In this very narrow temperature range the density of water can justifiably be assumed to change linearly with the temperature. The density for 20.3 °C can be determined as follows:

Using $t_2 - t_1 = 1$ °C, $\rho_2 - \rho_1 = -0.00043$ g/cm^3; $t - t_1 = 0.3$ °C, $\rho - \rho_1 = \rho - 0.99823$g/cm^3, and substituting into the interpolation equation $\frac{-0.00043}{1} = \frac{\rho - 0.99823}{0.3}$ is obtained. Solving this equation gives the **density of water at 20.3 °C: ρ = 0.99810 g/cm^3**

1. INTRODUCTION TO THE LABORATORY
SAFETY, LABORATORY EQUIPMENT, GAS BURNERS

Objectives

Overview of the general and safety rules of working in a chemical laboratory. Introduction to the most important laboratory equipment.

Introduction: Laboratory safety

Textbooks and the supplementary material provide abundant information on how to work in a chemical laboratory in a safe and scientifically sound manner. During the first lab session, the instructors will re-emphasize all the important points. However, simply knowing the rules is not enough to guarantee laboratory safety. It is the experimenters' duty and responsibility to work always in a way that does not endanger either themselves or others working in the same laboratory.

Pre-lab Assignment

Read the introduction chapter of this lab manual.

1.1. Safety Training

Date: ....

1. General safety rules in a chemical laboratory: (summarize the most important information briefly!)

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2. What are the most important rules of **fire safety** in a chemical laboratory?

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3. What are the relief measures for the following laboratory accidents?

	acid	**base**
poured over a hand	....	....
	....	....
splashed into eyes	....	....
	....	....
accidentally ingested	....	....
	....	....

4. List the most often used chemicals which pose a safety hazard and should be dealt with special care. What safety rules must be observed when working with these chemicals?

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Review Exercises and Problems

1. Are the following statements true (T) or false (F)?

A ..... Only injuries requiring treatment should be reported to your laboratory instructor.
B ..... Changes in laboratory procedures are encouraged without authorization to heighten student interest.
C ..... Working alone in the laboratory is *not* permitted under any circumstances.
D ..... Do not return excess, unused chemicals to the stock bottle.
E ..... Always wash your hands before leaving the laboratory.
F ..... Smoking, drinking, eating, and chewing are permitted outside of the laboratory only.
G ..... Before dispensing a chemical, read its label twice.
H ..... Always dispense twice as much chemical from a reagent bottle than the amount suggested in the experiment outline.
I ..... When mixing solutions, always add the dilute solution to the concentrated one.
J ..... Never taste a chemical.
K ..... Gas burners temporarily not in use should be adjusted to produce yellow, luminous flame.
L ..... Everyone must wear a labcoat in the laboratory at all times.

1.2. Laboratory equipment

Date: ....

1. List the most important pieces of laboratory equipment using the scheme below.

Glassware

heatproof

not heatproof

Porcelain equipment

Metal equipment

Wooden equipment

2. Draw the <u>cross section</u> of a beaker, an Erlenmeyer flask and a volumetric flask.

beaker

Erlenmeyer flask

volumetric flask

1.3. Use of Gas Burners

Date: ....

1. List the steps of installing and lighting a *Bunsen* burner:

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2. MASS AND VOLUME MEASUREMENTS

Objectives

To overview the metric and SI units of mass and volume measurements. To introduce the laboratory equipment used for mass and volume measurements. To demonstrate the concept of significant figures and its use in stating results of measurements. To introduce the concepts of precision and accuracy.

Pre-lab Assignment

Read the introduction of this lab manual.

2.1. Mass measurement – Analytical and standard laboratory balances

Pre-lab Exercises

*1. What is the definition of the **sensitivity** and **weighing capacity** of a balance?*

What are the usual properties of a standard laboratory balance?

weighing capacity:...................... sensitivity: ....

What are the usual properties of an analytical balance?

weighing capacity:...................... sensitivity: ....

2. a/ Why is the real mass of an object different from the mass measured in air? Which of these two quantities is smaller?

b/ What factors influence the magnitude of this error?

3. A certified 5-g (x) weight is weighed three times in order to calibrate three different standard laboratory balances. The results:

Measurement	*Balance 1 (g)*	*Balance 2 (g)*	*Balance 3 (g)*
1	*5.05*	*5.17*	*5.16*
2	*5.00*	*5.15*	*4.89*
3	*4.99*	*5.09*	*4.91*

a/ Calculate the difference (Δ) between the average mass ($\bar{x}$) and the real mass (x) for each set of data. (See Pages 3-4)

$\bar{x}_1 =$ $\Delta_1 = \bar{x}_1 - x =$

$\bar{x}_2 =$ $\Delta_2 = \bar{x}_2 - x =$

$\bar{x}_3 =$ $\Delta_3 = \bar{x}_3 - x =$

b/ Compare the different balances based on their accuracy (the difference between the average mass and the real mass).

c/ What is the mean deviation ($\bar{\delta}$) of the different balances? (See Pages 3-4)

$\bar{\delta}_1 =$

$\bar{\delta}_2 =$

$\bar{\delta}_3 =$

d/ Compare the precision of different balances.

Mass measurement – Analytical and standard laboratory balances

Date: ....

Experiment Outline

Select one of the standard objects provided by the instructors and weigh it using a standard laboratory balance. If you use a simple pan-balance, **record which certified weights you used and also the order in which you put them in the measuring pan.**

If you use a digital balance, make sure you use the balance correctly and figure out how taring works on the balance.

Weigh the same standard object using an analytical balance. If you use a simple pan-balance, record which certified weights you used during the measurement (in later laboratory work, recording only the mass is sufficient). **Make sure you can use the balance routinely and confidently for later work!**

1. List the most important features of standard laboratory balances (Summarize the most important properties based on the demonstration of the instructors.)

Weighing a standard object on a standard laboratory balance

Name of the object:...

Mass of the object: .... g

2. List the most important features of analytical balances (Summarize the most important properties based on the demonstration of the instructors.)

Weighing a standard object on an analytical balance

Name of the object:...

Mass of the object: .... g

Review Exercises and Problems

1. List the most often used SI and metric units of the following physical quantities (see Page 2)!

Quantity	SI unit	Metric unit	Conversion factor
length (l)			
volume (V)			
mass (m)			
amount of substance (n)			
density (ρ)			

2. Which unit is larger?

1 kg	....	1 g	1 dm^3	....	1 l
1 cm^3	....	1 ml	1 J	....	1 cal
1 mg	....	1 ng	1 Pa	....	1 bar
1 nm	....	1 mm	1 g/cm^3	....	1 t/m^3

2.2. Volume measurement

Date: ....

Experiment Outline

The instructors introduce the most important volume measuring devices, demonstrate their correct use and the proper method for preparing standard solutions. **If you have never used a volumetric flask or pipette before, practice their use by measuring water several times.**

1. Classify the most important volume-measuring devices:

- Not certified measuring devices:

- Certified measuring devices

calibrated for inflow		calibrated for outflow

2. Based on the demonstration list the most important steps

- of **using a volumetric pipette**

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Review Exercises and Problems

1. A chemist needs 10 cm^3 of a solution. What accuracy can be achieved using

a.) a measuring cylinder:................cm^3, b.) a burette:.................cm^3, c.) a pipette:.................cm^3.

2. A chemist needs 50 cm^3 of a solution (ρ= 1.000 g/cm^3). List the six different listed methods in the order of their decreasing accuracy:

a/ 50 cm^3 measured by a measuring cylinder

b/ a 50 cm^3 volumetric flask is filled up and its content poured out into a beaker

c/ a 50 cm^3 volumetric pipette used properly

d/ a 10 cm^3 volumetric pipette used properly five times

e/ a volume-graded beaker is filled up to the 50 cm^3 mark

f/ 50.0000 g solution is measured by an analytical balance

most accurate $\longrightarrow$ least accurate

....

2.3. Calibration of volumetric measuring equipment

Date: ....

Experiment Outline

The nominal volume of measuring devices is checked by weighing the water that fills (volumetric flask) or flows out of (pipette, burette) the device. Distilled water should be used for the calibration. Before the measurement, the water and all the devices used in the calibration should be kept in the balance room for some time to allow their temperature to reach that of the air in the room. Before the measurement, make sure that the temperature of air and the water used for calibration agree within ±0.5 °C.

***Calibration of pipettes and burettes*:** Determine the mass of the dry weighing dish together with its cap on an analytical balance. Record the temperature of water to a precision of 0.1 °C and fill the pipette or burette to the mark. Let the water flow out to the measuring dish and weigh it again.

Calibration of a volumetric flask: Weigh the empty and dry volumetric flask together with its stopper. Record the temperature of water, fill the volumetric flask to the mark and weigh it again.

Use the standard density table to find the density of water at the measured temperature. Use interpolation if necessary. Calculate the volume from the mass and the density.

Calibrate your pipette following the method described in the experimental outline!

Calibration of a Pipette

Type of the pipette to be calibrated (graduated, single-volume): ....

Temperature in the balance room: ....°C

Temperature of the water used: ....°C

Density of water at ....°C: ....g/cm^3

(Find the appropriate value in a table. Use interpolation if necessary.)

Nominal volume of water (V_n): ....cm^3
Mass of water (m): ....g

Measured volume of water: $\boldsymbol{V_\mathrm{m} = \frac{m}{\rho} =}$ **= .... cm^3**

Difference between the measured and nominal volumes in %:

$$\frac{V_\mathrm{m}-V_\mathrm{n}}{V_\mathrm{n}} \times 100 =$$

3. PREPARATION OF A STANDARD SOLUTIONS

Objectives

To overview the most important concentration units and their use to calculate the mass of solid necessary to prepare a standard solution. To practice the preparation of standard solutions.

Pre-lab Exercise:

Calculate the amount of solid necessary to prepare the solution!

Volume of solution to be prepared (V): ....

Concentration (c): ....

Formula of salt from which solution is prepared: ....

Amount of the salt to be used for preparing the solution: $n = c \times V =$

The molar mass of the crystalline salt: $M =$

The mass of solid to be weighed: $m = n \times M =$

Preparation of a standard solution from crystalline salt

Date: ....

Experiment Outline

The instructors will tell you the identity and concentration of the salt your solution should contain. Calculate the amount of solid necessary and prepare the solution by the proper method already demonstrated.

1. Based on the demonstration, list the most important steps of preparing a **solution** in a volumetric flask starting form a solid salt

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2. Preparation of a solution

- Weighing data (mass of the solid actually weighted):

- Steps of the solution preparation:

- Calculation of the **accurate** concentration of the solution (it can only be calculated after measuring the accurate weight of the solid used):

The mass of the solid weighed: m =

The molar mass of the crystalline salt: M =

Amount of the salt used for preparing the solution: $n = m/M =$

Concentration of the solution: $c = n/V =$

4. DENSITY MEASUREMENTS

Objectives

To introduce the density-measuring devices used in a chemical laboratory. To determine the density of the prepared solution and calculate the weight-percent composition based on the density.

Pre-lab Assignment

Find some background information about the following topics in a general chemistry textbook and read it: **density measurement**

Pre-lab Exercises

1. What methods are known for measuring density? List some device appropriate for measuring density!

2. Estimate whether the density of the following objects is larger or smaller than the density of water.

a.) a wooden cube of 1×1×1 m: ....,
b.) your body ....,
c.) a piece of limestone: ....,
d.) wood chips: ....,
e.) a small lead ball: ....

3. The density of water at room temperature is almost exactly 1 g/cm^3. A chemist wants to determine the density of water by measuring a 25-cm^3 sample. What precision (how many significant figures) should be quoted in the result if the following measuring devices are used

a/ an analytical balance and a 25 ml single-volume pipette: ....,
b/ a standard laboratory balance and a single-volume pipette: ....,
c/ an analytical balance and a measuring cylinder: ....,
d/ a standard laboratory balance and a measuring cylinder: ....
e/ an analytical balance and a pycnometer: ....

4. The density of a solid is determined by first weighing a piece on an analytical balance (41.7523 g). The volume of the same piece is measured (21.3 cm^3) in a measuring cylinder through the volume of displaced liquid. Give the density of the solid to the appropriate number of significant figures.

5. The density of solid **A** is 2.70 g/cm^3, that of solid **B** is 3.87 g/cm^3. A 1.00 g piece of each solid is placed into a measuring cylinder containing 5.00 cm^3 water. Which piece displaces more water? What is the difference between the displaced volumes of water (in cm^3)?

Density measurement using a pycnometer

Experiment Outline

1. Determination of the volume of the pycnometer

- Select a suitable pycnometer, make sure it is dry and weigh it on an analytical balance together with all its accessories (m_1). Record the temperature reading if the pycnometer has a built-in thermometer, otherwise record the laboratory temperature.
- remove the pycnometer from the balance room and fill it with distilled water with special care to avoid the formation of bubbles. Insert the thermometer or stopper into the pycnometer. If your pycnometer has a marked capillary ending, the level of water should rise above the mark. Drain excess water with filter paper or a dry piece of cloth from the outside of pycnometer and (if needed) from the top of the stopper or thermometer. Make sure that the outer surface of the pycnometer is dry.
- If your pycnometer has a marked capillary ending, drain the water from this capillary using a narrow piece of filter paper until the level of water reaches the mark. Handle the pycnometer very carefully and avoid holding it in your hands for periods longer than absolutely necessary because your hands are warmer than the pycnometer and heat may corrupt the measurement.
- Weigh the filled pycnometer (m_2), and record the temperature reading. If your pycnometer does not have a separate thermometer, measure the temperature of water separately after the measurement. If your pycnometer has a marked open-end capillary it may be difficult to weigh it consistently because evaporation of the relatively volatile solvent causes the mass to decrease in time. This phenomenon often poses a difficulty with water or aqueous solution. The best way to deal with this problem is to set the level of water a little above the mark, place the pycnometer on the balance, and record the mass when the level of water is exactly at the mark.
- Calculate the volume of the pycnometer after finding (and possibly interpolating) the density of water (ρ_w) at the measured temperature:

$$\text{volume of the pycnometer: } V = (m_2 - m_1) / \rho_w$$

2. Measurement of the mass of the solution in the pycnometer

Rinse the pycnometer with a small amount of your solution three times, fill it carefully with the solution in the same way you used for filling it with water. Make sure the outer surface is dry and weigh the pycnometer (m_3).

$$\text{mass of the solution} = m_3 - m_1$$

3. Calculation of the density of the solution

The density is calculated by dividing the mass of the solution by the volume of the pycnometer:

$$\rho = (m_3 - m_1) / V$$

4. Calculation of the weight percent composition of the solution

- The weight percent composition can be calculated from the concentration (obtained from the mass of solute and volume of solution) and density of the solution.

Density measurement using a pycnometer

Date: ....

1. Measurement of the density of the solution

The name of the salt from which the solution was prepared: ....

The exact concentration of the solution: ....

Weighing data:

Mass of the dry pycnometer (m_1) ....g

Mass of the pycnometer filled with water (m_2) ....g

Mass of the water in the pycnometer ($m_w=m_2-m_1$) ....g

Temperature of water:..............°C, density of water (ρ_w):................g/cm^3

(from the table, given to four significant digits)

Mass of the pycnometer filled with the sample (m_3) ....g

Temperature of the sample:..............°C

Calculate the volume of the pycnometer

$V = (m_2–m_1)/\rho_w =$ ....g/..................g/cm^3 = ...cm^3

Calculate the density of the sample:

$\rho = (m_3-m_1)/V =$ ....g/.................cm^3 = ... g/cm^3.

2. Determination of the weight percent composition of the solution

Calculate the mass of 100.00 cm^3 sample using the measured density (see above)

$m_{\text{solution}} = V\cdot\rho =$ .... g

Calculate the mass of solute (dissolved salt without crystallization water) in this solution

$m_{\text{salt}} =$ .... g

Calculate the weight percent composition of the solution:

$\frac{m_{salt}}{m_{solution}} \times 100 =$ = ... %(m/m).

5. SEPARATION OF MIXTURES, PURIFICATION OF SOLIDS

Objectives

Introduction to basic chemical laboratory operations: grinding, dissolving, decanting, centrifuging, filtration, crystallization. Separation of binary mixtures based on the different solubility of components, purification of the water soluble component by crystallization.

Pre-lab Assignment

5.1. Decantation, Centrifuging, Filtration

Pre-lab Exercise

1. How can we induce crystallization from the solution of a compound?

2. What are inclusions in a crystal? How can their formation be avoided during crystallization?

Decantation, Centrifuging, Filtration

Date: ....

Experiment Outline

The instructors demonstrate the use of different separation techniques.

1. What is the purpose of centrifuging a sample?

2. What is the purpose of decantation, when can it be used?

3. List the different types of filtration techniques and their area of laboratory use as shown in the demonstration.

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4. Draw the picture of a vacuum filtration apparatus fitted with a Büchner funnel and name its parts!

5.2. Purification of benzoic acid sample contaminated with sodium chloride

Date: ....

Experiment Outline

Weigh about 2 g of contaminated benzoic acid on a standard laboratory balance and dissolve it in ca. 15 cm^3 1:1 acetone:water mixture. After dissolution, filter the residue out on folded filter paper and rinse the beaker with ca. 5 cm^3 1:1 acetone:water mixture. Add ca. 80 cm^3 cold distilled water to the filtrate. Filter the precipitated crystals using a Büchner-funnel and an aspirator and wash them with ice-cooled distilled water.

The **purity** of benzoic acid can be **tested** as follows: collect the washing water going through the funnel in a test tube. Acidify it with some dilute nitric acid and add two drops of reagent solution of silver nitrate. White precipitation (AgCl) indicates a large amount of contamination, whereas a slightly turbid solution indicates a small amount of contamination. The solid is free of chloride only if the solution remains clear in this test after the addition of silver nitrate. Dry the purified chloride-free sample in air and weigh it.

Weighing of the contaminated sample:

Contaminated sample: .... g

Observations during the precipitation of benzoic acid:

Write down the ***chemical equation*** of the silver nitrate test for chloride ion.

Result of the silver nitrate test:

Weighing of purified benzoic acid:

Mass of benzoic acid obtained: .... g

Mass of contamination: .... g

The amount of ***contamination*** of the original sample of benzoic acid was .... % (m/m).

6. TEMPERATURE MEASUREMENT, CHANGES OF STATE

Objectives

A review of different methods used for temperature measurement. Boiling point and melting point as physical properties characteristic of a substance. Determination of the melting point of a solid and the boiling point of a liquid.

Introduction

The melting point of a solid is characteristic of its identity. The melting point is the temperature at which the solid and liquid phase of the substance are in equilibrium. Melting point does not usually depend sensitively on outside pressure, but is often markedly influenced by the presence of impurities.

Evaporation of liquids only proceeds at the surface at temperatures below the boiling point. The vapor pressure gradually increases when the temperature increases, and reaches the outside atmospheric pressure at a certain temperature. The liquid begins to boil at this point. Thus **the boiling point** is the temperature at which its vapor pressure equals the outside atmospheric pressure. The boiling point of substances depends sensitively on pressure. The higher the pressure, the higher the boiling point is. The boiling point referenced to an outside pressure of a 101325 Pa is usually called the **normal boiling point** and is listed in tables.

It may happen that the temperature of a liquid rises higher than the boiling point. This is called overheating. Overheating may corrupt the results of boiling point measurements. To avoid this error, substances known to induce boiling are often added to the liquid.

Pre-lab Assignment

Find some background information about the following topics in a general chemistry textbook and read it.

Gas burners
Temperature measurement
Melting point
Overcooling
Boiling point

Pre-lab Exercises

1. Draw the schematic picture of the normal (non-luminous) flame of a laboratory gas burner and give the approximate temperatures for each section.

2. Compare the normal and luminous flame of a laboratory gas burner. Which of these flames is hotter?

7. ACID-BASE TITRATIONS

Objectives

To prepare a standard solution of strong acid from a concentrated solution and determine its precise concentration by acid-base titration.

Find some background information about the following topics in a general chemistry textbook and read it.

- the principles of acid-base titrations in general
- the working principles of acid-base indicators

Pre-lab Exercises

1. List the safety rules concerning concentrated bases and their dilution:

2. 400.00 cm^3 ~0.04 mol/dm^3 NaOH solution needs to be prepared by the dilution of a concentrated NaOH solution (50 weight%, density: 1.53 g/cm^3). What is the volume (in cm^3) of the concentrated solution needed?

The amount of substance in the NaOH solution to be prepared:

c = 0.02 mol/dm^3,

V = 500 cm^3 = 0.500 dm^3

n(NaOH) = $c \times V$ = ……………. mol

The mass of this amount:

M(NaOH) =

m(NaOH) = $n \times M$(NaOH) = …………. g

100 g of the concentrated solution contains 50 g NaOH.

Calculate the mass of the concentrated solution that contains the necessary amount of NaOH:

m(concentrated solution) =……………. g

The volume of this solution:

$$V = \frac{m}{\rho} = \frac{\qquad\qquad\qquad \text{g}}{\frac{\text{g}}{\text{cm}^3}} = \textbf{…………….. cm}^3$$

What volume would you measure if you had only a 5 cm^3 graduated pipette with 0.1 cm^3 graduation marks as the best piece of volumetric equipment for this purpose?

7.1. Preparation of a standard solution of hydrochloric acid

Experiment Outline

Objective:

Preparation of ~100 cm^3 HCl solution of the required concentration (0.3-0.6 M) by diluting a concentrated solution of hydrochloric acid (38 weight%, ρ = 1.189 g/cm^3).

Procedure:

Calculate the volume of concentrated solution necessary in cm^3 using the known weight% and the density.

Put about 50 cm^3 of distilled water into a beaker and add the amount of concentrated HCl solution necessary using a small, plastic measuring cylinder. (**Never use a pipette for measuring concentrated acid solutions.**) **Be careful and avoid pouring drops of this concentrated solution onto anything.** Mix the liquids in the beaker and then add about 50 cm^3 of distilled water. Stir the solution well until the solution becomes entirely homogeneous. Store the homogeneous solution in a flask, and label it. Do not store the solution without a cap and open to the atmosphere.

7.2. Determination of the concentration of the standard hydrochloric acid solution

Pre-lab Exercises

1. 10.00 cm^3 0.183 mol/dm^3 HCl solution is exactly neutralized by 9.85 cm^3 unknown NaOH solution. What is the concentration of the NaOH solution? Give the result to a reasonable precision.

2. Check the transition pH interval of the acid-base indicators used in the following experiment in a textbook or other suitable book.

phenolphthalein: .... methylred: ....

Determination of the concentration of the standard hydrochloric acid solution

Date: ....

Experiment Outline

Dilute the prepared standard solution tenfold: measure 10.0 cm^3 standard solution with a single-volume pipette into a 100 cm^3 volumetric flask, fill up with water to the mark and shake the solution until it is entirely homogeneous.

Use the 10.0 cm^3 single-volume pipette to measure 10.00 cm^3 of the provided standard NaOH solution with known concentration (**record its concentration in your lab manual**) into 3 separate titration flasks or beakers. Dilute the solution to a final volume of 30-40 cm^3 with distilled water and add two or three drops of methylred indicator solution. Fill the burette with the *dilute* HCl solution. Titrate the first sample with constant stirring quickly until the indicator shows approximately the transition color. This will give an approximate value for the equivalent volume (V'). The second and third titrations should be done more carefully. The titrating solution can be added quickly until V = V'–0.5 cm^3 reached. The final increments should be added slowly, drop by drop until the exact transition color of the indicator is reached.

Calculate the average of the titrations (exclude the first, if you find the volume inaccurate!) and determine the precise concentration of the standard HCl solution.

Preparation of the dilute solution:

10.00 cm^3 of the previously prepared standard solution is diluted to 100.0 cm^3 and carefully homogenized.

Titration of the dilute HClsolution

volume of the titrated NaOH solution: .... cm^3

concentration of the titrated NaOH solution: .... mol/dm^3

volume of the HCl solution added until transition color is reached:

measurement 1 (approximate) .... cm^3

measurement 2 .... cm^3

measurement 3 .... cm^3

average: .... cm^3

Calculation of the precise concentration of the diluted HCl solution:

The precise concentration of the diluted HCl solution: .... mol/dm^3

The precise concentration of the original (10-times more concentrated) standard HCl solution:

.... mol/dm^3

Remarks (compare the intended and the actual concentration of your standard HCl solution)