

# FACULTY OF ENGINEERING DEPARTMENT OF ELECTRICAL AND ELECTRONICS ENGINEERING

# CHM1001 BASIC CHEMISTRY

# LABORATORY MANUALS



Doç.Dr. Miraç Nedim MISIR

Öğr.Gör.Dr. Sinan NOHUT

# MATERIALS USED IN CHEMISTRY LABORATORIES











Erlen

Beaker

Volumetric flask

Glass balloon

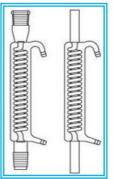
Graduated













Nuçe Erlen

Funnel

Quantitative funnel Separating funnel

Cooler

Rubber bulb



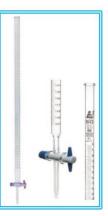
















Pliers











Metal-porcelain spatula

Test tube

Hour Glass

Piset







Porcelain crucible



Porcelain mortar



Buchner funnel











Bunsen burner Asbestos and tripod Metal tongs Wooden tongs

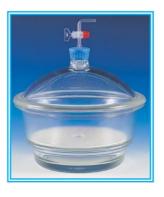








Desiccator



Vacuum desiccator



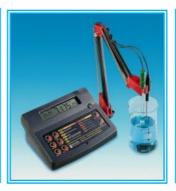
Sport



Protective gloves



Magnetic Stirrer



Benchtop pH meter



Precision scales







Oven Oven Water bath





Goggles

Rules to be observed while working in the laboratory





#### **Solutions and Their Preparations**

Solutions are single-phase (homogeneous) mixtures consisting of at least two different substances.

The most important feature that shows the chemical content **is concentration**. Concentration is expressed in various units.

It is a quantity that shows the relative amount of solute in a unit amount of solution or solvent.

Some of the concentration units are based on volume, some on mass, and some on the number of moles.

It is defined on the basis of. In order to find the equivalents of these units in terms of each other, the solute and the molar mass values of the solvent as well as the density of the solution are often sufficient to know. When preparing a solution with a known concentration on a volume basis, the solvent to be added to the solution

While the amount of solvent is not tal en into consideration, when preparing the solution in other concentration units, the amount

The amount of solvent must also be calculated.

Concentrations by Volume: Molarity (M), Normality (N), Mass % in Volume (w/v, w/v)

Concentrations by Mass: Percent mass (%a), Molality (m), ppt, ppm, ppb

Mole Concentrations: Mole percent and mole fraction (for physicochemical quantities)

Dilution of solutions can generally be defined as the solute/solvent ratio.

concentration means reducing the concentration by adding solvent, and concentration means decreasing the concentration.

It means to increase the amount of solvent added or mostly the vapor pressure is high.

can be removed by evaporation of the solvent.

Solutions on a volume basis are easy to dilute (M1  $\times$  V1 = M2  $\times$  V2), while solutions on a mass basis are easy to dilute (M1  $\times$  V1 = M2  $\times$  V2). In concentration units, there is much greater ease of preparation and no need for volumetric flasks. It has the convenience of not being heard.

## **Solution Preparation and Concentration Applications**

Frequently used in expressing solution concentrations;

molarity, normality, molality, % mass, % volume, % mass/volume, ppm, % mole and mole fraction.

In cases where the density of the solution needs to be known, a known volume is taken with a pipette.

It will be used to measure the mass of the solution by transferring it to a clean and dry beaker that has been previously tared or zeroed on an electronic scale, and the density is mass/volume.

will be remembered.













## **Concentration Units**

Definitions and definitions of concentration units commonly used to indicate the concentration of solutions.

The relevant equations are given below:

**1. Molarity** : It is the number of moles of the substance dissolved in 1 liter of solution.

M = nsolute / Vsolution (Liters)

2. Normality : It is the number of equivalent grams of the substance dissolved in 1 liter of solution.

 $N = nsolute \times T / Vsolution (Liters)$ 

3. Molality : It is the number of moles of the substance dissolved in 1 kg gram of solvent.

m = nsolute / msolvent (Kg)

4. Percentage by Mass : The amount of dissolved substance in grams in 100 grams of solution.

 $%(w/w) = (gsolute / g solution) \times 100$ 

**5. Percentage by Volume** : It is the volume amount of the substance dissolved in 100 mL of solution.

 $\%(v/v) = (Vsolute / Vsolution) \times 100$ 

6. Percentage by mass/volume: The amount of grams of the substance dissolved in 100 mL of solution.

 $%(w/v) = (gsolute / Vsolution) \times 100$ 

7. Mole Fraction : The number of moles of solute divided by the total number of moles of components in the solution.

is the ratio.

X = nsolute / ntotal

#### **SOLUTION PREPARATION**

A substance exists as molecules or ions in a second substance (usually water)

The homogeneous mixture formed when the water is dispersed is called **a solution** .

A substance that is dispersed into ions or molecules is called a solute; a substance that dissolves the substance .

The second substance is called **the solvent**. To indicate the amount of dissolved substance in the solution.

The term "concentration" is used for .









Figure 1. Solution preparation

Here we will examine three types of concentration:

- a. Percentage (%) concentration
- **b.** Molarity
- c. Normality
- **a. Percentage (%) Concentration:** It is div<u>ided into three groups</u> as mass, volume and mass/volume. leaves.
- 1) Concentration by mass (w/w): The amount of solute in g of solution. is the amount.

Experiment: Prepare 100 g of 5% NaCl solution by mass.

There should be 5 g of NaCl and 95 g of pure water in 100 g of this solution. Accordingly, 5 g of NaCl is weighed is transferred to a beaker, Erlenmeyer flask or volumetric flask. Add 95 g of pure water or 95 mL of pure water (water density d ÿ 1 g/mL) is added and mixed until the NaCl is completely dissolved.



2) Concentration by volume ( v/v): The amount of substance dissolved in 100 mL of solution . The amount is in mL.

Experiment: Prepare 50 mL of 10% ethanol solution by volume.

If we were to prepare 100 mL of solution, 10 mL of alcohol and 90 mL of pure water would be required. 50 mL If half of these amounts are taken as a solution, the solution is prepared. A measuring cylinder or a balloon 5 mL of alcohol and 45 mL of pure water are added to the volumetric flask.

3) Concentration by mass/volume (w/v): g of solute in 100 mL of solution is the amount as .

Experiment: Prepare 100 mL of 5% NaCl solution by mass.

There should be 5 g of NaCl in 100 mL of this solution. Accordingly, 5 g of NaCl is weighed and transferred to a 100 mL volumetric flask and dissolved by adding some pure water, then added to 100 mL again. is completed with pure water.

b. Molarity: The number of moles of the substance dissolved in 1 liter of solution.

Experiment: How to prepare 100 mL of 1 M NaCl solution? (mA NaCl = 23 + 35.5 = 58.5 g/mol).

$$M= \quad \frac{n}{V} = \quad \frac{\frac{m}{mA}}{V} \qquad \text{from the formula,}$$

$$1 = \frac{\frac{m}{58.5}}{0.1} \quad \ddot{y} \text{ m} = 5.85 \text{ g}$$

Since the solution is 100 mL, the amount of NaCl to be taken is 1/10 of the mole amount, i.e. 5.85 g.

Take NaCl and transfer it to a 100 mL volumetric flask. Add some pure water and mix completely.

is dissolved. Then it is completed with pure water up to the mark. The mouth of the volumetric flask is closed, mixed and labeled. Thus, 1 M 100 mL NaCl solution is prepared.

Experiment: How to prepare 250 mL of 0.2 M solution from 1 M NaCl solution?

This is a dilution process. For this, the following dilution equation is used.

#### V1 = 50mL

Take 50 mL of 1 M solution and complete it to 250 mL with pure water in a volumetric flask.

### **Preparation of Solutions from Liquids**

When preparing a solution from liquid substances (acid or base), first the molar ratio of that liquid is determined. concentration is calculated. Then, the relevant solution is prepared with the help of the dilution formula.

Experiment: How can a 0.10 M and 250 mL HCl solution be prepared from 36% HCl with a density of d=1.18 g/mL? prepared? (mA HCl = 1 + 35.5 = 36.5 g/mol)

### **Molarity Formula:**

First of all, the amount of pure substance is calculated. For this;

The formula for the amount of pure substance (m) = % ÿ d ÿ V is used (V=1000 mL is taken).

According to this formula; first the amount of pure HCl in this solution is found.

Amount of pure substance (m) = % ÿ d ÿ V

Number of moles of HCl (n) = 
$$\frac{m}{mA}$$
 =  $\frac{424.8g}{mA}$  = 11.6 moles

Molarity (M) = 
$$\frac{\text{mole}}{\text{Liter}} = \frac{11.6}{1.00L} = 11.6 \text{ mol/L (Molar = M)}$$



# **Another solution:**

The molarity calculated above can also be calculated with a single formula. For this, volume without consideration;

$$M = \frac{\% \times d \times 10}{m_A}$$

The formula can also be used. Similarly, when the data is substituted into the formula, direct molarity is found.

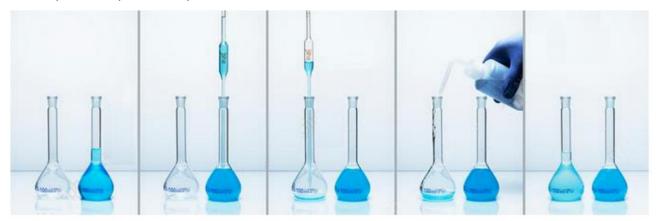
$$M = \frac{\% \times d \times 10}{m_{\Delta}} = \frac{36 \times 1,19 \times 10}{36.5} = 11,6 M$$

Then from the dilution equation;

M1 ÿ V1 = M2 ÿ V2

11.6 ÿ V1 = 0.10 ÿ 250 ÿ **V1 = 2.2 mL 2.2 mL** 

is taken from the first HCl solution with a pipette and transferred to a 250 mL volumetric flask and the volume is marked. It is completed with pure water up to the line.



c. Normality: It is the number of gram equivalents of the substance dissolved in 1 liter of solution.

Effect Valence (t): Number of protons (H+) in acids, number of ions (OH-) in bases,

is the number of positively charged ions.

$$N = \frac{m \times Tesir değerliği}{m_{A} \times V} \qquad N = M \times Tesir değerliği$$

Experiment: How to prepare 0.2 N 250 mL NaOH solution? (mA NaOH = 40 g/mol)

The effective valence of NaOH is t = 1.

Eşdeğer gram sayısı (veya ekivalent) = 
$$\frac{m_A}{Tesir değerliği}$$
 =  $\frac{40}{1}$  = 40

$$N = \frac{m \times t}{m_{\Delta} \times V} \Rightarrow m = \frac{N \times m_{\Delta} \times V}{t} = \frac{0.2 \times 40 \times 0.250}{1} \Rightarrow m = 2 g$$

Weigh 2 g of NaOH, transfer it to a volumetric flask and dissolve it with some pure water. Then, the volume is It is completed with pure water up to 250 mL (mark line).

## **QUESTIONS**

- 1. How do you prepare 500 mL of a 20% solution from an 80% alcohol solution?
- 2. If there is 60.376 g of KCI in a 450 g KCI solution, what is the percentage of this solution?
- 3. From 98% H2SO4 with d=1.89 g/mL,
  - a) 1000 mL of 2 M and 500 mL of 0.5 M
  - b) How do you prepare 1000 mL of 0.2 N and 100 mL of 0.4 N solution?

### PRECISION REACTIONS

Some substances dissolve very well in water, while others do not dissolve at all. Inorganic substances are divided into two groups: water-soluble and water-insoluble.

Water Soluble:

- All Lithium compounds (LiCl, LiF, Li2SO4 etc.)
- All Sodium compounds (such as NaCl, NaNO3, Na2SO4, Na2CO3, NaCH3COO, Na3PO4 ...)
   All Potassium compounds (such as KCl, KI, KNO3 ...)
   All Cesium compounds (such as CsCl, CsNO3 ...)
   (ammonium) compounds (such

as NH4Cl, NH4Br, NH4NO3, (NH4)2SO4 ...) • All NH4

• All Chlorate (CIO3 - ), perchlorate, (CIO4 - ), acetate (CH3COO- ) and nitrate (NO3 - ) compounds:

Ca(ClO3)2, Mg(ClO4)2, Pb(CH3COO)2, NH4CH3COO, Pb(NO3)2 ... etc. Also acid salts (HS- , HCO3 - (Sr(HS)2, Mg(HCO3)2,  $\,$ , HPO4  $\,$  -2, H2PO4 - )

CaHPO4, Ba(H2PO4)2 ... etc.) are soluble in water.

Insolubles in Water:

• Hg2Cl2, SrSO4, BaSO4, Ag2SO4, PbSO4, HgSO4. • Some

hydroxides and carbonates are also insoluble in water: Mg(OH)2, MgCO3, Ca(OH)2, CaCO3,

BaCO3, SrCO3, Al(OH)3, Fe(OH)3. • Other

than these, S-2 , SO3 <sup>-2</sup>, PO4 <sup>-3</sup>, CrO4 <sup>-2</sup> compounds (except for 1st Group cations and NH4 <sup>+</sup> compounds) are insoluble in water such as BaCrO4, Ca3(PO4)2, CoS, FeS, Cr2(SO3)3 ...

However, contrary to the solubility rules mentioned above; HgCl2, Ba(OH)2, Sr(OH)2 and BaS completely soluble in water.

Experiment: In this experiment, two different solutions will be mixed together to observe the "collapse" phenomenon.

BaCl2 solution is prepared in one test tube and Na2SO4 solution is prepared in the second test tube. One is placed on top of the other. is poured and mixed. A white, cloudy BaSO4 solution is observed. Here, the 2Na+ and 2Cl- ions are not shown in the net ionic equation of the reaction since they do not undergo any change. Accordingly, the net ionic equation is as follows:

Similarly, prepare individual solutions of other substances and mix them with each other.

Observe whether precipitation occurs and write the net ionic equations.

After preparing these solutions and mixing them, observe whether there is any precipitation and Write the net ionic equations in your notebook.

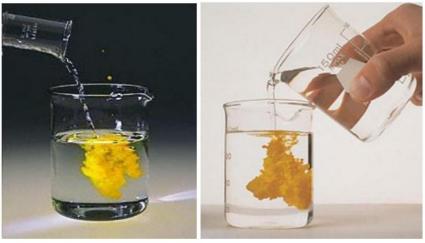




Figure 2. Precipitation of PbI2

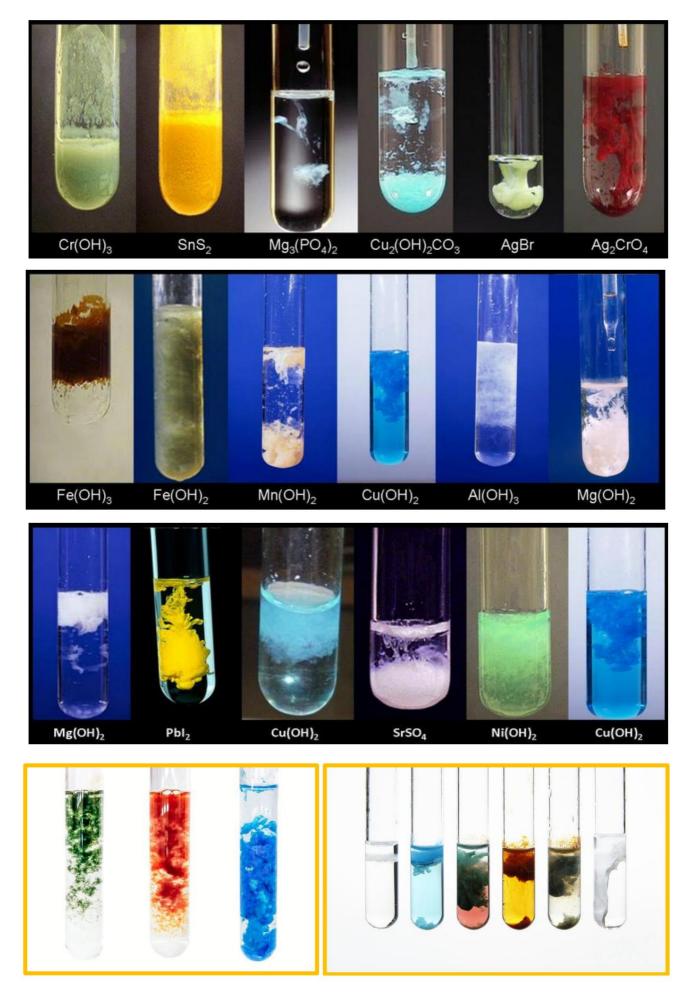


Figure 3. Precipitation colors of some cations.

### SIMPLE DISTILLATION (DISTILLATION)

The temperature at which the vapor pressure of a liquid equals the external pressure is called **the boiling point** of that liquid. The boiling point of a liquid depends on the external pressure. If the external pressure is reduced, the boiling point also decreases. Usually, the pressure should also be stated when indicating the boiling point. For example; water boils at 100°C at 760 mm Hg pressure.

The boiling point of a compound also depends on the molecular weight of the compound and the intermolecular forces of attraction. If more heat is given to a liquid at its boiling point, the temperature of the liquid does not increase. However, the heat given causes the liquid to turn into vapor, and the temperature remains constant until the liquid is completely removed as vapor.

As explained above, liquids turn into vapor with the help of heat, and vapor turns into vapor again. The process of purifying a liquid by condensing it is called **distillation**.

Boiling point is a characteristic physical property for purity control of liquids.

Experiment: A simple distillation apparatus is set up as shown below. After a solution of liquids with different boiling points or a solid-liquid solution is placed in the distillation flask, a few boiling stones are thrown into the flask. Then, a thermometer is attached to the mouth of the flask using a cork. After a reflux condenser is attached to the other end of the flask, the flask is slowly heated.

During this time, the temperature should be constantly checked with a thermometer. In liquid-liquid solutions, the liquids with lower boiling points are first distilled and separated. In solid-liquid solutions, the solvent is distilled and separated at the boiling point.

Boiling points of some liquids:

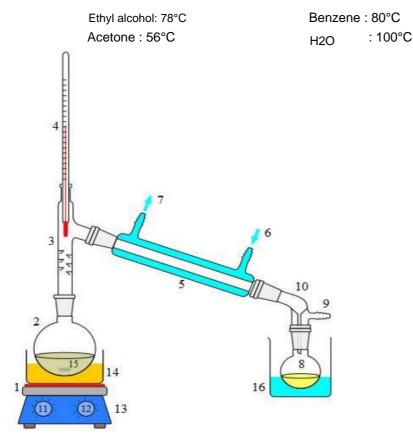


Figure 4. Simple distillation setup

- 1: Hot plate
- 2: Distillation flask
- 3: Distillation neck
- 4: Thermometer
- 5: Cooler
- 6: Water inlet
- 7: Water outlet
- 8: Collection balloon
- 9: Vacuum outlet
- 10: Alonge
- 11: Temperature control
- 12: Mixer control
- 13: Heater / mixer
- 14: Heating bath
- **15:** Magnets or boiling stones
- 16: Cooling bath

# **FLAME TRIALS**

Some of them are separated by precipitation and cannot be easily recognized (in terms of color etc.) Flame tests are used, which are more decisive for cations.

Electrons belonging to metal cations excited in the flame move to high energy empty orbitals.

They do (absorption, absorption). From this unstable state, they return to their old orbitals.

rotating electrons emit the energy they have received in the form of rays (emission, radiation). These rays wavelengths are different for each element. If these wavelengths are in the visible region (~400-800 nm)

In the flame experiment, these rays create colors that can be observed with the naked eye. More than one element When they are together, the rays can interfere with each other and the wavelength of some rays filters and spectroscopic devices are used because they may be outside the visible region. This is called spectral In the experiment conducted to identify metal cations, the color formation mechanism in the flame is seen in detail in Figure 5.

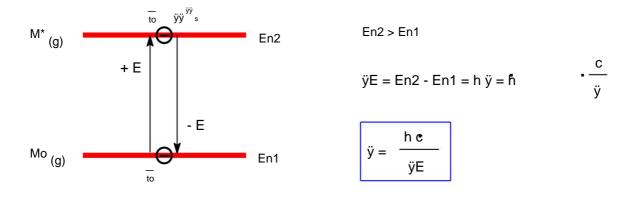


Figure 5. Flame mechanism of metal cation solutions.

Experiment: Salts containing cations (Ba+2, Sr+2, Cu+2 etc.) to be tested for flame recognition

Aqueous solutions are prepared and transferred to spray containers. Aqueous solutions are sprayed onto the flame the colors formed are observed.

The colors produced by some cations in the flame test are given below:

Na+: Yellow Li+: Carmine red

Sr+2 : Fez red K + : Violet

Ba+2 : Yellow-green Ca+2 : Brick red

Materials required: Bunsen burner, cobalt glass, KCI, NaCI, LiCI, BaCI2, CaCI2, SrCI2



Figure 6. Colors given off by some cations in the flame.

### **ACID - BASE REACTION AND pH DETERMINATION**

#### **Definitions:**

Acid: Briefly, they are substances that give H+ ions to their aqueous solutions .

**Base:** Substances that give OH-ions to their aqueous solutions.

**Indicators:** These are substances used to determine the turning point.

5 mL of the given sample (HCI) is taken with a pipette and transferred to a conical flask.

Add 50 mL of pure water and a few drops of phenol phthalein and mix with 0.2 M NaOH in the burette.

titration is stopped when the solution in the Erlenmeyer flask turns a permanent pink color. At this point, the Erlenmeyer flask

The acid in it is completely neutralized by the added base. This point is called the turning point .

The volume of 0.2 M NaOH consumed up to the end point is read from the burette (V1).

The molarity is found using the following equation:

M1  $\ddot{y}$  V1 = M2  $\ddot{y}$  V2 are substituted into the formula and the unknown M2 (HCI concentration) is calculated.

$$(M1 = 0.2; V2 = Read; M2 = ?; V2 = 5 mL)$$

In pH determination, **pH = - log[H** | formula is used.

How to do the experiment: Take 4 test tubes. Add HCI, HNO3, H2SO4 and

NaOH is added. **Methylorange is added to the acidic ones, and phenolphthalein** is added to the basic ones. Color transformations are noted.





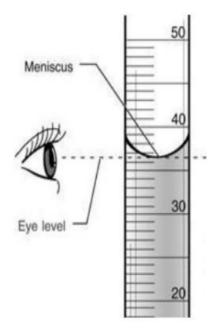
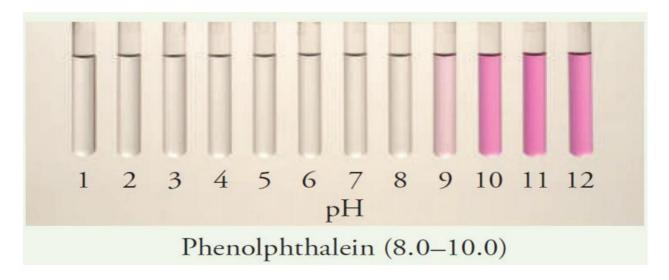
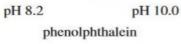


Figure 7. Titration apparatus and reading of meniscus



Color change of phenol phthalein indicator with pH change





Phenol phthalein



pH 3.2 pH 4.4 methyl orange

Methyl orange

pH = -log [hidrojen iyonu derişimi]

Asidik Nötr Bazik

0 1 2 3 4 5 6 7 8 9 10 11 12 13 14

# **EXPERIMENT REPORT SAMPLE**

Faculty: Section: No: No: Name Surname: Experiment No: Name of the Experiment:	Date of Experiment
o Explanations on the experiment and its procedure in your laboratory handout	
o Important explanations you learned from your laboratory supervisor	
o Your observations during the experiment	
o Calculations, if any.	