<div align="center">EXPERIMENT</div>

Conductivity of electrolyte solutions

Goal

The terms of **conductivity** and **electrolytes** will be introduced and related to the concept of dissociation. The use of the conductivity meter, very similar to the pH meter, will help to differentiate between strong and weak electrolytes. In the last part of the experiment, the presence of chemical reactions will be identified by measuring changes in conductivity.

Materials

- ☐ jumbo test tubes
- ☐ conductivity meter
- ☐ 6M solution of NH_3
- ☐ 6M solution of CH_3COOH
- ☐ Ethyl alcohol

- ☐ 0.1M solutions of KNO_3, KCl, KOH, HCl, and $Ca(NO_3)_2$
- ☐ an unknown solution
- ☐ 5mL, 10mL, 25mL pipets (preferred Mohr's)
- ☐ 250mL and 50mL beakers
- ☐ rinsing water bottle

Background

Playing with water near electrical appliances can be risky. Surprisingly, however, water does not conduct electricity. For electricity to propagate through media there must be free charges that move along the electric field. Pure water does not contain a significant number of charges and does not conduct electricity. However, when there is something dissolved in the water, therefore making an aqueous solution, it can be conductive.

Conductivity and aqueous solutions

Whether the aqueous solution is conductive or not depends on the nature of the solute. If the solute dissociates into ions when dissolved in water, the ions will conduct electricity. These types of chemicals are called electrolytes. If the solute dissolves as a molecule without dissociation, the solution will not conduct electricity and the solute will be called nonelectrolyte.

Conductivity units

The unit for conductivity is Siemens per centimeter, $S{\cdot}cm^{-1}$. Pay attention to the units during this experiment because the tool will often display prefixes like milliSiemens or microSiemens per centimeter. Temperature affects conductivity and must be recorded during the measurement of this property.

Types of electrolytes and factors affecting their conductivity.

Salt and sugar are soluble in water. When sugar dissolves in water its molecules remain intact because water can not break apart the strong covalent bonds between their atoms. As a neutral molecule, sugar in solution will not conduct electricity and it is an example of a **nonelectrolyte**. Salt, sodium chloride or NaCl, is an ionic compound that gives place to two ions, Na^+ and Cl^- when dissolved in water. These ions are responsible for the solution's conductivity. The number or more specifically the concentration, and the mobility of these ions will determine how conductive the solution is. Based on these principles we can classify substances as:

× **Non-electrolytes**. When the aqueous solution of that substance does not conduct electricity. In general terms, these electrolytes present low conductivities, lower than $10\mu S \cdot cm^{-1}$.

× **Weak electrolyte**. When the aqueous solution of that substance conducts electricity poorly. In general terms, these electrolytes present medium conductivities, between $10\text{-}1000\mu S \cdot cm^{-1}$.

× **Strong electrolyte**. When the conductivity of the aqueous solution of that substance is high. In general terms, these electrolytes present large conductivities, larger than $1000\mu S \cdot cm^{-1}$.

The **mobility** of the ions is related to their size. Small ions move fast which enhances conductivity, while large ions encounter more resistance to move and show lower conductivities. The table below displays a series of mobility values for positive and negative ions at infinite dilution and 298K and 1 atm.

Ion	H_3O^+	Li^+	Na^+	Mg^{2+}	OH^-	Cl^-	Br^-	NO_3^-
movility, u^∞ $(10^5 cm^2 \cdot V^{-1} \cdot s^{-1})$	363	41	52	55	206	80	81	74

The effect of ions' mobility is easily appreciated in isomolar solutions, which are solutions with the same molarity. The more ions in the solution, the higher the conductivity will be. For the same substance, the number of ions is determined by the **concentration**; more concentration means more ions and more conductivity. It is important to recall that only ions contribute to conductivity. While some molecules dissociate completely, others dissociate only partially. The latter are weak electrolytes. Interestingly, when two solutions containing ions are mixed, the conductivity of the resulting solution will be equal to the sum of the two separate solutions with the same concentration as in the mixture. This is called the **additivity rule** and will hold as long as the ions do not react with each other. When two electrolytes undergo a chemical reaction, new products will form and the additivity rule will not apply. A large deviation from the sum of separate conductivities is an indication of a chemical reaction.

Example

The conductivity of a 0.050 M solutions of HNO_3 is measure to be 19.6 mS/cm. That of a 0.050 M solution of KCl is 9.6 mS/cm. A new solution is prepared mixing 10 mL of HNO_3 0.10 M and 10 mL of KCl 0.10 M. The conductivity of the resulting mixture is measure to be 29.6 mS/cm. Calculate the concentration of each spices in the mixture and determine if their compounds have reacted or not.

Answer: To calculate the new concentration use the formula for dilutions:

$$V_1 \times M_1 = V_2 \times M_2$$

where V_1 and M_1 are the initial volume and concentration and V_2 and M_2 are the values after the dilution.
We look for M_2, therefore

$$M_2 = V_1 \times \frac{M_1}{V_2}$$

for HNO_3,

$$M_2 = \frac{10\,mL \times 0.10\,M}{(10\,mL + 10\,mL)} = 0.05\ M$$

The same values apply for KCl.
We can see that the addition of the separate conductivities (κ) is very close to the total conductivity of the mixture with the same concentrations for each electrolyte.

$$\kappa(HNO_3\ 0.05\ M) + \kappa(KCl\ 0.05\ M) = 19.6\ mS/cm + 9.6\ mS/cm = 29.2\ mS/cm$$
$$\kappa(HNO_3\ 0.05M + KCl\ 0.05M) = 29.6\ mS/cm$$

The additivity rules applies, meaning that there is no reaction between HNO_3 and KCl.

Procedure

Part A. Getting started: standardize the conductimeter

☐ *Step 1:* – Install the conductivity meter. The probe of the instrument is immersed in a clean solution that must be kept aside until the end of the experiment. Mind the probe needs to be hydrated at all times.

☐ *Step 2:* – To calibrate the conductivity meter, place 40mL of standard conductivity solution in a 50mL beaker while placing the probe in it.

☐ *Step 3:* – Make sure the conductivity cell is fully submerged in the liquid.

☐ *Step 4:* – Now press Setup (top right menu). Normal setups are: cell constant of 1.0, standard recognition with manual selected, reference temperature 25 degrees, temperature coefficient of 2.1%, and alarm limits off.

☐ *Step 5:* – Now press Standardize (top right menu) and input 1.000 mS/cm (or 1000 μS/cm) in the keypad and press confirm (this should be the conductivity value of the standard). Finally, press Confirm (top right menu) to save the standard value. Now you are ready to measure.

Part A. Getting started: measuring conductivity

☐ *Step 1:* – It is very important to follow some cleaning steps before you measure the conductivity of any solution. You will need a 100 mL beaker labeled as waste, a wash bottle, and a 250 mL beaker with distilled water.

1. Rinse the probe using the wash bottle over the waste beaker.
2. Dip the probe 3-4 times into the 250 mL beaker with clean distilled water.
3. Dip the probe into the solution to be measured. Use a 50 mL beaker (or a jumbo test tube) with at least 25 mL of solution. Use the same volume for all conductivity measurements.
4. Repeat cleaning steps 1 and 2.

☐ *Step 2:* – It is critical to properly read the conductivity units from the conductimeter. Every solution would give a different conductivity value and sometimes a different unit. The conductivity units are written next to the measurement in the conductivity. It is critical you copy the measurement with the unit given by the tool.

> **Good Lab Practice**
>
> ✍ To ensure the probe is well cleaned and the water in the 250 mL beaker remains clean, the conductivity meter should display a value close to that of the distilled water during the cleaning.
>
> ✍ If the conductivity value of the cleaning water differs from the one of distilled water, replace the water in the 250 mL beaker with fresh distilled water, and thoroughly clean the probe.

Part B. Pure substances.

☐ *Step 1:* – Following the procedure outlined above, measure the conductivity of distilled water. Record the value in the Results section.

☐ *Step 2:* – Following the procedure outlined above, measure the conductivity of tap water. Record the value in the Results section.

☐ *Step 3:* – Following the procedure outlined above, measure the conductivity of Ethyl alcohol (C_2H_5OH). Record the value in the Results section.

☐ *Step 4:* – Classify the substance as strong, weak, or nonelectrolyte.

Part C. Series of electrolytes.

☐ *Step 5:* – In a clean 50 mL beaker, add 20 mL of 0.10 M HCl and 20 mL of distilled water (this is a $\frac{1}{2}$ dilution). Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers. Mix well and measure the conductivity. Recalculate the concentration after the dilution and write it down in the Results section.

☐ *Step 6:* – In a clean 50 mL beaker, add 20 mL of 0.10 M KOH and 20 mL of distilled water (this is a $\frac{1}{2}$ dilution). Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers. Mix well and measure the conductivity. Recalculate the concentration after the dilution and write it down in the Results section.

☐ *Step 7:* – In a clean 50 mL beaker, add 20 mL of 0.10 M KCl and 20 mL of distilled water (this is a $\frac{1}{2}$ dilution). Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers. Mix well and measure the conductivity. Recalculate the concentration after the dilution and write it down in the Results section.

☐ *Step 8:* – In a clean 50 mL beaker, add 20 mL of 0.10 M KNO_3 and 20 mL of distilled water (this is a $\frac{1}{2}$ dilution). Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers. Mix well and measure the conductivity. Recalculate the concentration after the dilution and write it down in the Results section.

☐ *Step 9:* – In a clean 50 mL beaker, add 20 mL of 0.10 M $Ca(NO_3)_2$ and 20 mL of distilled water (this is a $\frac{1}{2}$ dilution). Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers. Mix well and measure the conductivity. Recalculate the concentration after the dilution and write it down in the Results section.

☐ *Step 10:* – Find an unknown solution. In a clean 50 mL beaker, add 20 mL of the unknown solution and 20 mL of distilled water (this is a $\frac{1}{2}$ dilution). Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers. Mix well and measure the conductivity.

☐ *Step 11:* – Classify all substances as strong, weak, or nonelectrolytes and identify the unknown compound based on the conductivity values. Know that the unknown can also be water.

Part D. Series of Dilutions.

☐ *Step 12:* – Obtained the conductivity of a 0.1 M solution of HCl from previous section. Write down the result in the Results section.

☐ *Step 13:* – Now you are going to prepare two dilutions. Starting from the concentrated 0.1 M solution of HCl, you are going to prepare 40mL volumes of the following dilutions: 0.050 M and 0.020 M. The first is a $\frac{1}{2}$ dilution, that is 20ml of acid needs to be mixed with 20ml of water to get 40mL of 0.050M solution, whereas the second is a $\frac{1}{5}$ dilution, that is 8ml of acid needs to be mixed with 32ml of water. Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers.

☐ *Step 14:* – Measure the conductivity of the two diluted HCl solutions (0.050 M, and 0.020 M) and record the values.

Part E. Mixtures of HCl and KNO_3.

☐ *Step 15:* – Write down the conductivity of the 0.050 M solution of HCl from the previous section in the Results section.

☐ *Step 16:* – Write down the conductivity of the solution of KNO_3 from the previous section in the Results section.

☐ *Step 17:* – In a clean 50 mL beaker, add 20 mL of 0.10 M HCl and 20 mL of 0.10 M KNO_3. Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers. Mix well and measure the conductivity. Recalculate the concentration after the dilution and write it down in the Results section, minding that both solutions have now the same concentration and therefore only one number needs to be listed.

Part F. Mixtures of NH_3 and $HC_2H_3O_2$.

☐ *Step 18:* – Prepare the following dilutions: 100mL of acetic acid ($HC_2H_3O_2$) 0.12 M from 6M (add 2mL of concentrated solution and fill up with water until 100mL) and 40mL of ammonia (NH_3) 0.12 M from 6M (add 2mL of concentrated solution and fill up with water until 100mL). Carefully read the concentration of the initial concentrated solutions indicated in the labels. Work in the hood when handling highly concentrated solutions.

☐ *Step 19:* – In a clean 50 mL beaker, add 20 mL of the 0.12 M NH_3 dilution and 20 mL of distilled water (this is a $\frac{1}{2}$ dilution). Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers. Mix well and measure the conductivity. Recalculate the concentration after the dilution and write it down in the Results section. Work in the hood when handling highly concentrated solutions.

☐ *Step 20:* – In a clean 50 mL beaker, add 20 mL of 0.12 M $HC_2H_3O_2$ and 20 mL of distilled water (this is a $\frac{1}{2}$ dilution). Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers. Mix well and measure the conductivity. Recalculate the concentration after the dilution and write it down in the Results section.

☐ *Step 21:* – In a clean 50 mL beaker, add 20 mL of 0.12 M NH_3 and 20 mL of 0.12 M $HC_2H_3O_2$. Use a Mohrs pipet to transfer the liquids, making sure you rinse with distilled water between transfers. Mix well and measure the conductivity. Recalculate the concentration after the dilution and write it down in the Results section, minding that both solutions have now the same concentration and therefore only one number needs to be listed.

⚠ CAUTION!

⚠ Handle concentrated acid or bases with care to avoid chemical burns.

⚠ Are you wearing your goggles? Do you know where the eye-washer is?

STUDENT INFO

Name: Date:

Pre-lab Questions

Conductivity of electrolyte solutions

1. Starting with a 6 M solution of NaCl, calculate the volume of solution and the volume of water necessary to prepare 10 mL of a 3 M solution of NaCl.

2. How can you dilute a 3 M solution to form a 1.5 M solution? (e.g. I would pick up X mL of the solution and YmL of distilled water.)

3. How can you prepare 40mL of acetic acid ($HC_2H_3O_2$) 0.12 M from 6M.

4. How can you prepare 40mL of ammonia (NH_3) 0.12 M from 6M.

5. Given the data below, classify the following electrolytes as strong, weak, or nonelectrolytes:

	Concentration (M)	Conductivity	Units	Type of electrolyte
$CuSO_4$	0.05M	4000	$\mu S \cdot cm^{-1}$	_____
$FeCl_3$	0.05M	10000	$\mu S \cdot cm^{-1}$	_____
$C_6H_{12}O_6$	0.05M	9	$\mu S \cdot cm^{-1}$	_____
CH_3COOH	0.05M	320	$\mu S \cdot cm^{-1}$	_____

Results
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Conductivity of electrolyte solutions

Part B: Pure substances

	Conductivity	Units	Electrolyte type
Distilled water	——————	———	———
Tap water	——————	———	———
C_2H_5OH	——————	———	———

Part C: Electrolytes

	Concentration (M)	Conductivity	Units	Electrolyte type
HCl	——————	——————	———	———
KOH	——————	——————	———	———
KCl	——————	——————	———	———
KNO_3	——————	——————	———	———
$Ca(NO_3)_2$	——————	——————	———	———
Unknown		——————	———	

Unknown #	——————	Unknown identity	———	

Part D: Concentration effect

	Concentration (M)	Conductivity	Units
HCl	_____	_____	____
HCl	_____	_____	____
HCl	_____	_____	____

Part E: HCl+KNO$_3$ mixture

	Concentration (M)	Conductivity	Units
HCl	_____	_____	____
KNO$_3$	_____	_____	____
HCl+KNO$_3$	_____	_____	____

Part F: HC$_2$H$_3$O$_2$+NH$_3$ mixture

	Concentration (M)	Conductivity	Units
HC$_2$H$_3$O$_2$	_____	_____	____
NH$_3$	_____	_____	____
HC$_2$H$_3$O$_2$+NH$_3$	_____	_____	____

STUDENT INFO

Name: Date:

Post-lab Questions

Conductivity of electrolyte solutions

1. Based on your results on Part B, which conductivity was higher, the one for distilled water or for tap water? Why?

2. Based on your results on Part C, specifically the conductivity values of KCl and KOH, how does ion mobility affect conductivity (see table in the Background section)? Use your data to illustrate your answer.

3. Based on your results on Part D, how does concentration affect conductivity? Use your data to illustrate your answer.

4. Based on your results in Part E, determine if their compounds have reacted or not. Does the additivity rule hold for this case?

5. Based on your results in Part F, determine if their compounds have reacted or not. Does the additivity rule hold for this case?

EXPERIMENT

Softening of hard water

Goal

The goal of this experiment is to understand the concepts of hard and soft water and their implications in daily life. The student will also experiment with the *dissociation of ionic compounds* in water, from which the **hydrogen-donor** nature of the acids and the notion of *pH* are emphasized. Other principles, such as equilibrium and ion exchange, are also presented.

Materials

- ☐ 250mL and 50mL beakers
- ☐ cation-exchange resin
- ☐ 6M HCl
- ☐ distilled water (not tab water)
- ☐ Litmus paper
- ☐ 50mL graduated cylinder
- ☐ 125mL Erlenmeyer

- ☐ NH_3/NH_4Cl buffer
- ☐ 0.01M $Na_2H_2($EDTA
- ☐ indicator (Eriochrome black mixed with NaCl)
- ☐ dropper solutions (0.10 M $Ca(NO_3)_2$, 0.10 M Na_2CO_3, and 0.10 M $Mg(NO_3)_2$)
- ☐ 0.10 M$NaHCO_3$ solution
- ☐ solid $CaCO_3$

Background

The term **"hard water"** refers to the concentration of certain minerals in the water. This topic concerns **aqueous solutions** in which water is the solvent and minerals are the solutes. These minerals are ionic compounds that dissociate in aqueous solutions producing ions. The hardness of water is an indication of the concentration of Ca^{2+} and Mg^{2+} ions.

Dissolving the insoluble

Ca^{2+} and Mg^{2+} ions originate in limestone ($CaCO_3$, the material of the pyramids) and chalk ($MgCO_3$, used to write on the chalkboard) deposits. However, chalk will not dissolve in water and neither the pyramids dissolve in the rain. How is dissolving these carbonates possible?

The answer is in the air

Water in contact with the air absorbs CO_2 in the atmosphere according to the equation:

$$CO_2 \text{ (g)} + H_2O \text{ (l)} \longrightarrow H_2CO_3 \text{ (aq)}$$
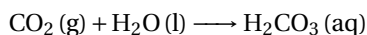
Where H_2CO_3 is carbonic acid, which reacts with the carbonate to form hydrogen bicarbonate. For the case of $CaCO_3$

$$CaCO_3 \text{ (s)} + H_2CO_3 \text{ (aq)} \longrightarrow Ca(HCO_3)_2 \text{ (aq)}$$
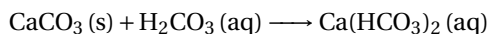
Calcium hydrogen carbonate is also called calcium bicarbonate. Bicarbonates are soluble in water (baking soda is sodium bicarbonate, $NaCO_3$) and, as ionic compounds, they are dissociated into its ions;
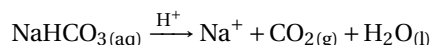
$$Ca(HCO_3)_2 \text{ (aq)} \longrightarrow Ca^{2+} + 2\,HCO_3{}^-$$
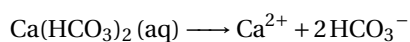
The overall reaction is the 3 previous equations added together:

$$CaCO_3 \text{ (s)} + CO_2 \text{ (g)} + H_2O \text{ (l)} \longrightarrow Ca^{2+} + 2\,HCO_3{}^-$$
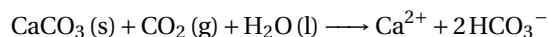
and this is how carbonates dissolve in water.

Sodium hydrogen carbonate can decompose under different conditions. On one hand, in acidic conditions, it produces carbonic acid that quickly decomposes to give carbon dioxide:

$$NaHCO_{3\,(aq)} \xrightarrow{H^+} Na^+ + CO_{2\,(g)} + H_2O_{(l)}$$

On the other side, when heated it produces: $2\,NaHCO_{3\,(aq)} \xrightarrow{\Delta} Na_2CO_3 + CO_{2\,(g)} + H_2O_{(l)}$

Implications in daily life

The presence of these ions in water has several implications for routine actions in our lives. The problem arises when the latter reaction takes place in the opposite direction, releasing CO_2 gas in the process and depositing solid carbonate. This direction is favored when water is heated and explains the white residue in bathrooms and kettles. This is especially a problem for boilers, that suffer from scaling when operating hard water.
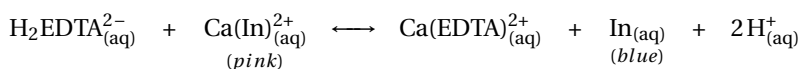
The experiment

The experiment consists of four parts: preparing the ion exchange resin, measuring the hardness of a sample of water, reducing the hardness also called softening, of the sample, and testing the hardness of the softened sample. Additionally, a series of simple reactions will be done to test the reactivity of Ca^{2+} and Mg^{2+} ions.

Measuring the hardness of the sample

The concentration of Ca^{2+} ions, or any other metallic cation, is usually measured by titration of the aqueous sample using a sodium salt as the titrant, a solution of known concentration added to the sample. First, an indicator will be added to the sample which will color the solution pink. The indicator chosen is Eriochrome Black T, which will turn blue when the Ca^{2+} ion is removed from the cation-indicator complex:

$$\underset{indicator\ complex\ (pink)}{Ca(In)^{2+}_{(aq)}} \longrightarrow Ca^{2+}_{(aq)} + \underset{indicator\ (blue)}{In_{(aq)}}$$
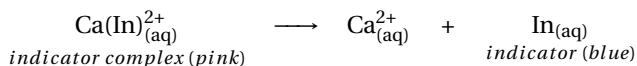
To achieve the color change H_2EDTA^{2-} will be used. H_2EDTA^{2-} (dihydrogen ethylenediaminetetraacetate) is an anion with a stronger affinity for the Ca^{2+} cations than the indicator. As H_2EDTA^{2-} is added to the solution it will replace the indicator forming a new complex, leaving the indicator alone, which in turn will become blue.

$$H_2EDTA^{2-}_{(aq)} + \underset{(pink)}{Ca(In)^{2+}_{(aq)}} \longleftrightarrow Ca(EDTA)^{2+}_{(aq)} + \underset{(blue)}{In_{(aq)}} + 2\,H^+_{(aq)}$$

Even when the example described above refers to calcium ions, the same chemistry applies to magnesium ions. It is important to note down the exact number of drops required to achieve the endpoint of the titration.

Softening the hard water

After measuring the hardness of the water sample, the next step is to soften the water. *Softening* is the process where ions, particularly calcium and magnesium ions, are removed from the water. When a hard water sample is softened we can refer to it as *softened water*. A cation-exchange resin will be used to soften the water. This resin contains many sulfonic groups, $(-SO_3H)$. Every two hydrogens in these acidic groups will be replaced by a metallic cation, Ca^{2+} and Mg^{2+}, in a mechanism called *cation exchange*. Finally, it is necessary to evaluate whether the water sample has been softened. As such the titration performed in Part A will be repeated, but this time with the softened sample. The most relevant parameter is the amount of EDTA added to achieve the endpoint of the titration, to compare this result to the previous titration. This experiment aims to be a qualitative analysis of the water treatment. For that reason, and to simplify the process, the titration will be done using droppers instead of the more precise burette typically used in titrations.

Procedure

Part A. Preparing the resin.

☐ *Step 1:* – Obtain around 5g of the dry, ion exchange resin in a 250 mL beaker. Remember that this resin can be recycled. Do not discard it at the end of the experiment.

☐ *Step 2:* – Add enough 6 M HCl to cover the resin. By adding the acid, the resin is cleaned of metallic cations. To use the ion exchange resin, it must be clean, also known as "in H^+ form".

> ⚠ CAUTION!
>
> ⚠ Handle concentrated acid with care to avoid chemical burns.
>
> ⚠ Are you wearing your goggles? Do you know where the eye-washer is?

☐ *Step 3:* – Now you are going to wash the acid away. After 2 minutes, add 200 mL of distilled water to the resin-acid mixture. Allow the resin to settle in the bottom of the beaker.

☐ *Step 4:* – Carefully, pour the liquid off (decant) into another beaker. This liquid is an HCl solution and should be handled and discarded as such.

☐ *Step 5:* – To ensure that all the acid is gone, wash again the resin with 200 mL of distilled water. Allow the resin to settle in the bottom of the beaker and pour the liquid into another beaker.

☐ *Step 6:* – Before you discard the liquid you can test the pH. Dip the tip of a glass stirring rod in the liquid and poke the wet end of the rod into a piece of Blue Litmus paper. If the Litmus paper turns pink, there is still acid, repeat steps 5 and 6. If it does not turn pink, the resin is clean and ready to be used. Reserve the resin for Part B.

Part B. Measure the hardness of a sample of softened water

☐ *Step 1:* – Obtain a little bit more than 150 mL of hard water in a beaker. Hard water is already prepared in the lab.

☐ *Step 2:* – Using a graduated cylinder, add 20.0 mL of hard water to a 125 mL Erlenmeyer. Reserve this water for Part C. Use the rest of the water for this part of the experiment.

☐ *Step 3:* – Transfer the remaining hard water to the beaker with the resin and let it stand for 25 minutes. In the meantime, proceed with Parts C and D.

☐ *Step 4:* – Pour off about 40 mL of the water sample that has been in contact with the resin into a clean beaker. This sample would be referred to as the softened water.

☐ *Step 5:* – Using a graduated cylinder, add 20.0 mL of the softened water to a 125 mL Erlenmeyer.

☐ *Step 6:* – Add 5 mL of an NH_3/NH_4Cl buffer solution (not to be confused with $Na_2H_2(EDTA)$) to the Erlenmeyer and the tip of a spatula (less than a pea-sized amount) of the indicator (a solid mixture of eriochrome black T and sodium chloride). Adding too many indicators will ruin the experiment. The solution color will be rose pink after the addition of the indicator.

☐ *Step 7:* – Now, we are going to titrate the water sample. Obtain about 20 mL of 0.01 M $Na_2H_2(EDTA)$ (the full name is Ethylenediaminetetraacetic acid) in a 50 mL beaker and a medicine dropper (or a plastic Pasteur pipette). Add the $Na_2H_2(EDTA)$ drop by drop to the mixture while stirring. As the endpoint is approached the solution color will become lavender. Count the drops needed for the analyte to turn blue. When the addition of one drop turns the solution blue, all Ca_2^+ and Mg_2^+ ions have been removed from the sample

☐ *Step 8:* – Clean the resin as described in Part A, and return the resin to the instructor–there is a large beaker ready to collect wet resin.

Part C. Measure the hardness of a water sample

☐ *Step 1:* – While Part B takes place, and the water becomes softer, add 5 mL of a NH_3/NH_4Cl buffer solution (not to be confused with $Na_2H_2(EDTA)$) to the Erlenmeyer, and the tip of a spatula (less than a pea-sized amount) of the indicator. Rememer that the indicator is just a solid mixture of eriochrome black T and sodium chloride. Adding too many indicators will ruin the experiment. The solution color will be rose pink after the addition of the indicator.

☐ *Step 2:* – Now, we are going to titrate the water sample. Obtain about 20 mL of 0.01 M $Na_2H_2(EDTA)$ in a 50 mL beaker and a medicine dropper (or a plastic Pasteur pipette). Add the $Na_2H_2(EDTA)$ drop by drop to the mixture while stirring. As the endpoint is approached the solution color will become lavender. Count the drops needed for the analyte to turn blue. When the addition of one drop turns the solution blue, all $Ca_2{}^+$ and $Mg_2{}^+$ ions have been removed from the sample

Part D. Observing reactions with Ca^{2+} and Mg^{2+} ions

☐ *Step 1:* – Obtain 4 test tubes and locate the following solutions: 0.10 M $Ca(NO_3)_2$, 0.10 M Na_2CO_3, 0.10 M$NaHCO_3$, and 0.10 M $Mg(NO_3)_2$.

☐ *Step 2:* – In test tube 1, add 10 drops of **$Ca(NO_3)_2$**. Add 10 drops of Na_2CO_3 and write down your observations.

☐ *Step 3:* – In test tube 2, add 10 drops of **$Ca(NO_3)_2$**. Add 2 drops of HCl and write down your observations.

☐ *Step 4:* – In test tube 3, add 10 drops of **$Ca(NO_3)_2$**. Add 10 drops of 0.1 M $NaHCO_3$ and write down your observations.

☐ *Step 5:* – Now, Heat test tube 3 in the flame of a Bunsen burner. Write down your observations.

☐ *Step 6:* – Now, cool down test tube 3. Add 2 drops of 6M HCl and write down your observations.

☐ *Step 7:* – In test tube 4, add a pea-sized amount of solid $CaCO_3$. Add 2 drops of 6M HCl and write down your observations.

☐ *Step 8:* – Now, repeat the steps above using **$Mg(NO_3)_2$** instead of **$Ca(NO_3)_2$**.

☐ *Step 9:* – At this point, complete the remaining steps of Part B.

Pre-lab Questions

Softening of hard water

1. A student needs 10 drops of EDTA to reach the equivalency point when titrating a sample of hard water. After the students soften the water with the resin, it requires 12 drops of EDTA to reach the equivalency. Are these results correct? Explain.

2. Find the definition of the following concepts:

    (a) Titration

    (b) Titrant

    (c) Litmus paper

    (d) Ion exchange resin

3. Where is the term **hard water** derived from?

4. Compare the sizes used in the experiment. Circle the bigger size:

    (a) Pea-size

    (b) Tip of a spatula

5. In the experiment, find the chemical name of EDTA.

STUDENT INFO

Name: Date:

Results
EXPERIMENT

Softening of hard water

Parts B and C: Comparing titrations

Indicate how many drops you used to achieve the endpoint for:

Hard water sample (Part C): _____   drops of Na_2H_2(EDTA)

Softened water sample (Part B): _____   drops of Na_2H_2(EDTA)

Part D: Observing reactions with Ca^{2+} and Mg^{2+} ions

Write down your observations such as gas evolution, the appearance of precipitate, dissolving of precipitate, and no observation.

	$Ca(NO_3)_2$	$Mg(NO_3)_2$
Na_2CO_3		
HCl		
$NaHCO_3$		
$NaHCO_3$+heat		
$NaHCO_3$+heat+HCl		
Solid $CaCO_3$+HCl		

Post-lab Questions

Softening of hard water

1. Based on your measurements. Did the ion exchange resin soften the hard water sample? Explain.

2. Write the balanced equations for the chemical reactions below. Mind that calcium carbonate is an insoluble compound.
.

$$Na_2CO_{3\,(aq)} \quad + \quad Ca(NO_3)_{2\,(aq)} \quad \longrightarrow$$

$$Ca(NO_3)_{2\,(aq)} \quad \xrightarrow{H^+}$$

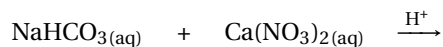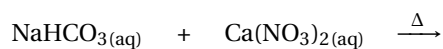$$NaHCO_{3\,(aq)} \quad + \quad Ca(NO_3)_{2\,(aq)} \quad \longrightarrow$$

$$NaHCO_{3\,(aq)} \quad + \quad Ca(NO_3)_{2\,(aq)} \quad \xrightarrow{\Delta}$$

$$NaHCO_{3\,(aq)} \quad + \quad Ca(NO_3)_{2\,(aq)} \quad \xrightarrow{H^+}$$

$$CaCO_{3\,(s)} \quad \xrightarrow{H^+}$$
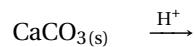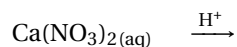
3. Write the balanced equations for the chemical reactions below. Mind that magnesium carbonate is an insoluble compound.

$$Na_2CO_{3\,(aq)} \quad + \quad Mg(NO_3)_{2\,(aq)} \quad \longrightarrow$$

$$Mg(NO_3)_{2\,(aq)} \quad \xrightarrow{H^+}$$

$$NaHCO_{3(aq)} \quad + \quad Mg(NO_3)_{2(aq)} \quad \longrightarrow$$

$$NaHCO_{3(aq)} \quad + \quad Mg(NO_3)_{2(aq)} \quad \xrightarrow{\Delta}$$

$$NaHCO_{3(aq)} \quad + \quad Mg(NO_3)_{2(aq)} \quad \xrightarrow{H^+}$$
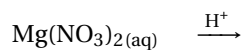
4. Using the equivalency between drops and mL (1mL=15drops), calculate the number of mL of $Na_2H_2(EDTA)$ needed for the titration of the soft and hard water samples.

<div align="center">

EXPERIMENT

Freezing point depression

</div>

Goal

The goal of this laboratory is to put into practice the theory seen for *properties of solutions* and to experimentally see the *freezing point depression* effect. The added value is to play detective by relating the nature of *colligative properties* to the molar mass, to identify the unknown powder.

Materials

- ☐ A ring stand and two iron rings with a wire gauze
- ☐ two or three jumbo text tubes with stoppers
- ☐ A series of 50mL, 100mL, 250mL and 600mL beaker
- ☐ Cyclohexane

- ☐ 20mL pipet
- ☐ Ice
- ☐ Perforated stoppers with stirring wire and thermometer

Background

During winter, salt is poured over the street to melt the ice on the floor. General knowledge says that the salt melts the ice, while some venture to claim that the salt lowers the freezing temperature of the water. Both assertions might be in the right direction but are, if not false, at least incorrect. When salt is mixed with water, there is no water or salt anymore. Instead, there is a *solution* of salt in water. Since the freezing point of the solution is lower than that of the pure water, the solution is no longer solid at outdoor temperatures. Notice that there is no need to state the nature of the solution or the solute because it is irrelevant. The depression of the freezing point is not a property specific to the salt, (i.e. the sodium chloride, NaCl), but rather a general effect of any solute. Salt is used because it is widely available, dissolves fast, and is inexpensive.

Freezing point depression

The formula governing the change (Δ) in the freezing point (T_f) is given by:

$$\Delta T_f = - K_f m$$

where m is the concentration in terms of *molality* and K_f is the *molal freezing-point depression constant*. K_f is characteristic of the solvent being experimentally determined. Remember that molality m refers to the moles of solute concerning the kilograms of solvent. ΔT_f is the difference between the freezing point of the pure solvent ($T_f^{solvent}$) and that of the solution ($T_f^{solution}$). The solvent used in this experiment will be *cyclohexane*, a very *volatile* organic solvent. Special precautions must be taken to avoid evaporation of the solvent. K_f for cylohexane is 20.1 °C/m. The solute will be an unknown compound. Hence you will not know its molar mass and therefore the molarity will not be explicitly known here.

Example

The freezing point of pure benzene is 5.50 °C. A solution is prepared by dissolving 0.450 g of an unknown substance in 27.3 g of benzene. The new freezing point is determined to be 4.18 °C. What is the molar mass of the unknown substance? The freezing point constant, K_f, for benzene is 5.12 °C/m.

Answer: calculate the concentration first:

$$\Delta T_f = K_f \times m \qquad m = \frac{\Delta T_f}{K_f} \qquad m = \frac{5.50°C - 4.18°C}{5.12°C\frac{kg}{mol}} = 0.258\frac{mol}{kg}$$

then, calculate the molar mass:

$$0.258\frac{mol}{kg} \times 27.3g \times \frac{1kg}{1000g} = 7.04 \times 10^{-3}\,mol \qquad \text{molar mass} = \frac{0.450g}{7.04 \times 10^{-3}\,mol} = 63.9\frac{g}{mol}$$

The experiment

The experiment today is divided into the following parts: finding the freezing point of the pure solvent, finding the freezing point of the solution, finding the freezing point after increasing the concentration, and solving the molar mass of the solute.
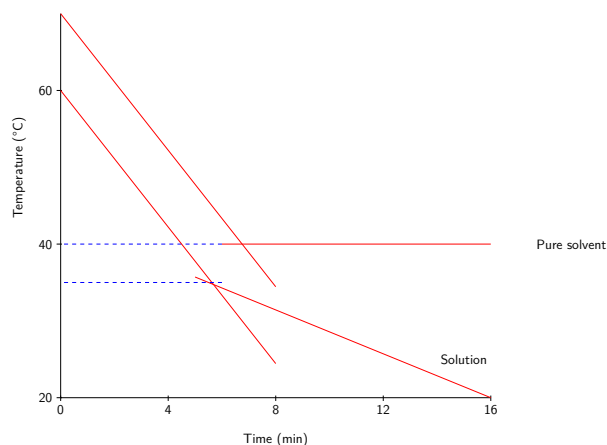
Cooling plots

During a phase change, all the energy transferred is used to reorganize the molecules and none is used to change the temperature of the substance. A liquid at a higher temperature than its surroundings gives up the heat and its temperature falls until it reaches the freezing point. The liquid continues to give up heat to the surroundings, but its temperature remains constant. Only when all the liquid is frozen will the temperature start to fall again.

If one plots temperature vs. time for the freezing of the pure liquid, a negative slope line will represent the cooling down, while the flat horizontal line (plateau) corresponds to the freezing process. These types of plots are called cooling plots. Notice that the transition from the cooling line to the horizontal line is not sharp. For that reason, the best way to determine the freezing point is to draw a straight line fitting most of the cooling points and another straight line fitting most of the cooling points, while finding the crossing point between two lines. You can measure the freezing point as the temperature measurement of this crossing point. For a pure solvent, the freezing point should be near the temperature corresponding to the plateau. This is the graphical method to find freezing points. In the case of a solution, this constant-temperature plateau does not exist anymore. In contrast, when a solution freezes its temperature decreases with time. However, the same graphical method can be used to obtain the freezing point of a solution. Mixing the solution well is critical to obtaining a good cooling plot. If you do not mix the solution well you will obtain a series of steps when the solution freezes that will not allow you to measure an accurate freezing point.

Finding the freezing point of the pure solvent.

In this part of the experiment, you will obtain the cooling plot of the pure solvent, cyclohexane. Using the graphical method you will obtain the freezing point as an average between two replications. You must plot the results carefully to obtain accurate results.

Finding the freezing point of a solution.

In this part of the experiment, you will obtain the cooling plot of a solution made of cyclohexane and an unknown solute. Using the graphical method you will obtain the freezing point. You will work with two different concentrations and will carry out one replication per concentration. This part aims to calculate the molar mass of the solute.

Example

Use the date replorted below to graph a cooling plot, and use the graphical method to calculate the freezing point of the substance. Is this a pure substance or a solution?

t (min)	T (°C)	t (min)	T (°C)
1	65.6	9	40
2	61.6	10	40
3	56.7	11	40
4	52.2	12	40
5	47.8	13	40
6	43.3	14	40
7	38.9	15	40
8	40	16	40

Answer: Based on the plot below, we have that at early times the liquid is being cooled down and its temperature decreases with time. At late times, the liquiz freezes and its temperature reaches a plateau. The graphical method to obtain freezing points consist on ploting a line for early times and another line for late times. These two lines intersect in a point, and the temperature for this point is the freezing point. As the temperature reaches a platea, we are dealing here with a pure substance.
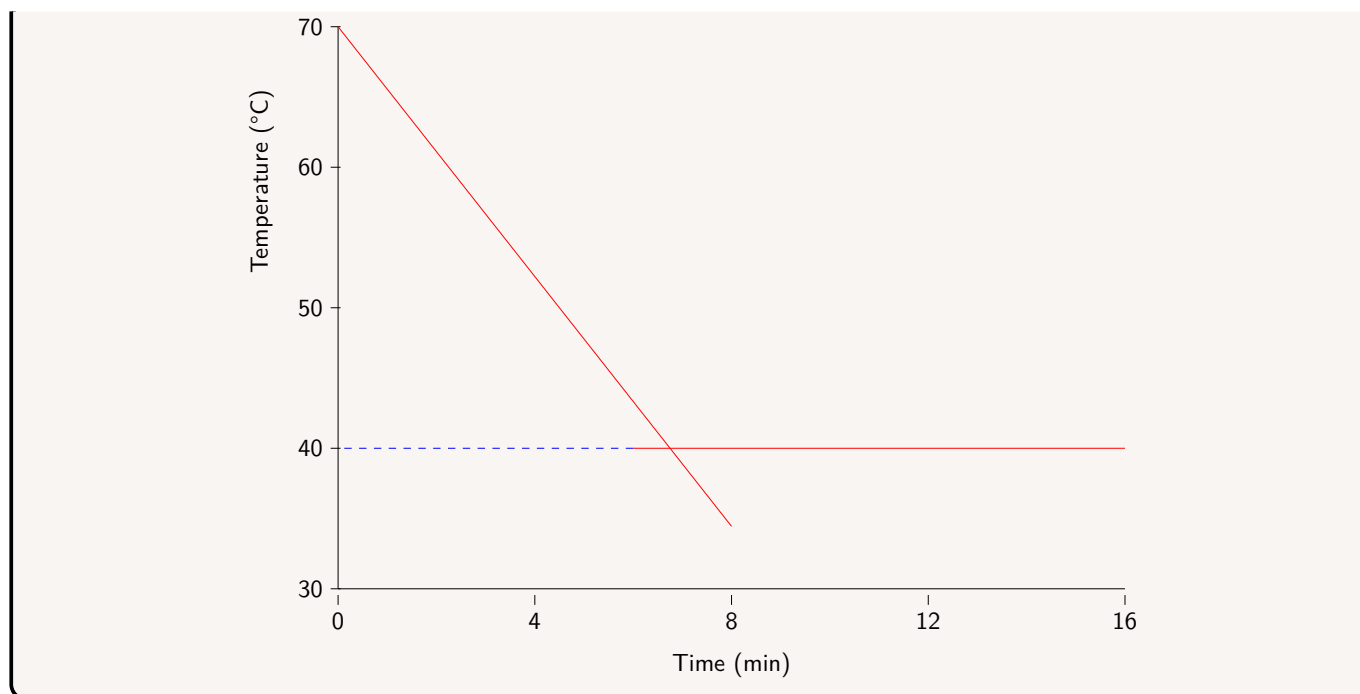
Procedure

Preparing the setup.

☐ *Step 1:* – Prepare the set-up to cool down the solution. Set up a ring stand and an iron ring with wire gauze on top. The height of the platform will normally be very low.

☐ *Step 2:* – Use a second, larger ring above the platform as protection. Attach a small clamp above the second iron ring.

☐ *Step 3:* – Put ice with some water in a 500 mL beaker and set it on the platform. This will be your cool bath. Water helps create a homogeneous bath.

> **Good Lab Practice**
>
> ✍ Never add the liquid or solid to be weighed to the container while on the scale. Take the container *off* the scale prior to adding the product. A spill on the scale will ruin both your measurement and the scale. ✍

Preparing a cyclohexane sample.

☐ *Step 1:* – Find 100mL and 50mL beakers and a 20 mL pipet.

☐ *Step 2:* – Obtain a large test tube that is clean and dry, a suitable rubber stopper, and a 100 mL beaker. The beaker will be used to hold the test tube vertically. Get the mass of the test tube with stopper and the beaker in the scale and write it down in the Results section.

☐ *Step 3:* – Pour no more than 25 mL of cyclohexane into the 50 mL beaker. Using the pipet, transfer exactly 20.0 mL of cyclohexane from the 50 mL beaker to the test tube and close it with the stopper immediately. Mind that as the liquid is volatile the measured volume will not be accurate. Using the scale get the mass of the test tube with the cyclohexane, the stopper, and the beaker. Make sure you do not weigh the thermometer or the stirring system.

☐ *Step 4:* – Get the mass of the filled-test tube with stopper and the beaker in the scale and write it down in the Results section.

Part A. Freezing point of the pure solvent.

☐ *Step 1:* – Insert the test tube into the ice and hold it vertically using the clamp. Make sure all liquid is fully submerged in the cool bath. Find the perforated stoppers with the stirring wire and the thermometer. The round wire end must encircle the thermometer probe. Exchange the stopper on the test tube with this gadget.

☐ *Step 2:* – As soon as the temperature is 15°C (or lower) start stirring the solvent with the wire and record the temperature every 15 seconds. Use all the digits given by the thermometer down to the tenth of the degree Celsius.

☐ *Step 3:* – Start plotting the results on the graph. Plotting temperature (in °C) in the vertical axis and time in the horizontal axis.

☐ *Step 4:* – Stop the experiment when the temperature starts dropping after several minutes of having a constant temperature. Do not stop until the temperature reaches at least 7°C.

☐ *Step 5:* – Now you will repeat the measurement one more time.

☐ *Step 6:* – Lift the test tube with the clamp above the ice and allow the cyclohexane to melt. Avoid using your hands to accelerate the process. Before the cyclohexane gets to 15°C get a second measurement by repeating the step above.

> **Good Lab Practice**
>
> ✍ Stir, and do not shake. It is very important to stir the cyclohexane constantly and gently until it is completely frozen.
>
> ✍ Make sure the wire stays around the thermometer and that the latter is centered in the test tube.
>
> ✍ The thermometer should not touch the walls of the test tube.
>
> ✍ A bad stirring method will lead to a partial freezing and the graph will not show straight lines.

Part B. Freezing point of the less concentrated solution.

☐ *Step 1:* – Pick up an unknown solute. You have to carry out Parts B and C at the same time.

☐ *Step 2:* – The same cyclohexane can be used in Part B. However, if you work in teams you can prepare another cyclohexane sample and carry out work in parallel.

☐ *Step 3:* – If you use the sample from part A, lift the test tube with the clamp above the ice and allow the cyclohexane to melt. Avoid using your hands to accelerate the process. During the next steps be careful to avoid the cyclohexane's temperature from getting above 15°C.

☐ *Step 4:* – If you prepare a new cyclohexane sample, see the steps above.

☐ *Step 5:* – Tare the scale with the weighing boat and obtain between 0.10 g and 0.11 g of the unknown solute. Remove the boat with the solute, tare the empty scale, and record the mass of the weighing boat together with the solute. Use the balance of maximum precision.

☐ *Step 6:* – Carefully add the solute into the test tube with the pure solvent. Make sure no solute is spilled. There is no need to add all the solute from the weighing boat, the amount added will be calculated by difference.

☐ *Step 7:* – Weight the weighing boat with the solute leftovers. Record the mass on the results page.

☐ *Step 8:* – Mix the solution well and insert the test tube in the ice while holding it vertically using the clamp.

☐ *Step 9:* – Start stirring the solution with the wire and record the temperature every 15 seconds. This time the temperature should not reach constant. Stop the experiment when the temperature reaches 1°C.

Part C. Freezing point of the more concentrated solution.

☐ *Step 1:* – Lift the test tube with the clamp above the ice and allow the cyclohexane to melt. Avoid using your hands to accelerate the process. During the next steps be careful to avoid the cyclohexane getting above 15°C.

☐ *Step 2:* – The same boat can be reused without cleaning. Tare the scale with the boat inside. Obtain between 0.24 g and 0.25 g of the unknown solute and record the mass of the weighing boat together with the solute using maximum precision.

☐ *Step 3:* – Add carefully the solute into the test tube with a fresh new 20mL sample of the pure solvent. Make sure no solute is spilled. There is no need to add all the solute from the weighing boat, the amount added will be calculated by difference.

☐ *Step 4:* – Weight the weighing boat with the solute leftovers. Record the mass on the results page.

☐ *Step 5:* – Mix the solution well and insert the test tube in the ice while holding it vertically using the clamp.

☐ *Step 6:* – Start stirring the solution with the wire and record the temperature every 15 seconds. This time the temperature should not reach a constant. Stop the experiment when the temperature reaches 0.5°C.

Calculations

(1) This is the mass of the empty test tube with a cork and placed in a beaker.

(2) This is the mass of the test tube filled with cyclohexane, with a cork, and placed in a beaker.

(3) This is the mass of solvent, m_{liquid}:

$$m_{solvent} = \boxed{2} - \boxed{1}$$

(4) This is the volume of cyclohexane added, where d is the density of cyclohexane:

$$v_{solvent} = \frac{m_{liquid}}{d} = \frac{\boxed{3}}{0.779}$$

(5) These are two replicates for the freezing point of the solvent obtained through the graphical method.

(6) This is the freezing point of the solvent.

(7) This is the mass of the weighing boat with solute before adding the solute to the solvent.

(8) This is the mass of the weighing boat with solute after adding the solute to the solvent.

(9) This is the mass of solute:

$$m_{solute} = \boxed{7} - \boxed{8}$$

(10) These are the freezing points of two solutions.

(11) These are the values of the freezing points depression:

$$\Delta T_f = \boxed{10} - \boxed{6}$$

(12) These is the molar mass of the solute:

$$\text{MW} = -\frac{K_f \cdot m_{solute}}{\Delta T_f \cdot m_{solvent}} \times 1000 = -\frac{20.1 \cdot ⑨}{⑪ \cdot ③} \times 1000$$

STUDENT INFO

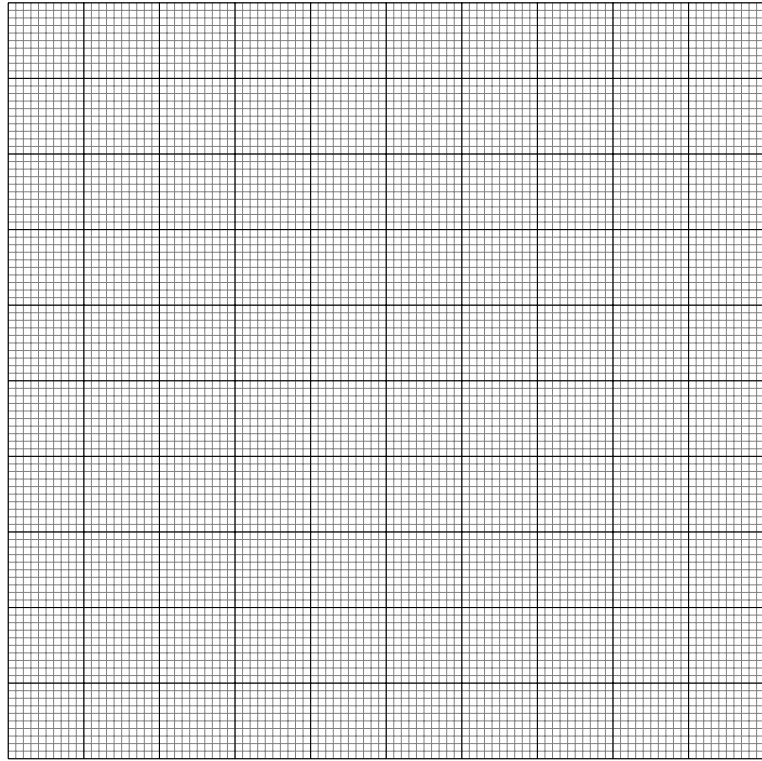Name: Date:

Pre-lab Questions

Freezing point depression

1. What does volatile mean?

2. What are the colligative properties?

3. What is molality? What are the units?

4. Why is molality used in this experiment instead of Molarity?

5. Use the date reported below to graph a cooling plot and use the graphical method to calculate the freezing point of the substance. Is this a pure substance or a solution?

t (min)	T (°C)	t (min)	T (°C)
1	55.6	9	30
2	51.1	10	28.6
3	46.7	11	27.1
4	42.2	12	25.7
5	37.8	13	24.3
6	33.3	14	22.86
7	28.9	15	21.6
8	31.4	16	20

(the question continues on the back)

$t_f \quad =$

Results
EXPERIMENT

Freezing point depression

Parts A. Freezing point of the pure solvent.

① Mass of the empty test tube with cork and beaker: —————— g

② Mass of the filled test tube with cork and beaker: —————— g

③ Mass of the cyclohexane: —————— g

④ Volume of the cyclohexane used: —————— mL

Trial 1

Time (sec)	Temp. (°C)	Time (sec)	Temp. (°C)
————	————	————	————
————	————	————	————
————	————	————	————
————	————	————	————
————	————	————	————
————	————	————	————

Trial 2

Time (sec)	Temp. (°C)	Time (sec)	Temp. (°C)
————	————	————	————
————	————	————	————
————	————	————	————
————	————	————	————
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(5) Freezing point of the pure cyclohexane. Trial 1: ⎯⎯⎯ °C   Trial 2: ⎯⎯⎯ °C

(6) Mean: ⎯⎯⎯ °C

Parts B and C. Freezing point of solutions.

①   Mass of the empty test tube with cork and beaker: _____ g

②   Mass of the filled test tube with cork and beaker: _____ g

③   Mass of the cyclohexane: _____ g

Low concentration	High concentration

Unkown #:   _____

⑦   Mass of boat: _____ g   Mass of boat: _____ g

⑧   Mass of boat (after adding):   _____ g   Mass of boat (after adding):   _____ g

⑨   Mass of solute: _____ g   Mass of solute: _____ g

Time (sec)	Temp. (°C)	Time (sec)	Temp. (°C)	Time (sec)	Temp. (°C)	Time (sec)	Temp. (°C)
____	____	____	____	____	____	____	____
____	____	____	____	____	____	____	____
____	____	____	____	____	____	____	____
____	____	____	____	____	____	____	____
____	____	____	____	____	____	____	____
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———— ————   ———— ————   ———— ————   ———— ————

	Low concentration		High concentration

(10) Freezing point: _____ °C   Freezing point: _____ °C

(11) ΔT_f: _____ °C ΔT_f: _____ °C

(12) Molar mass: _____ g/mol   Molar mass: _____ g/mol
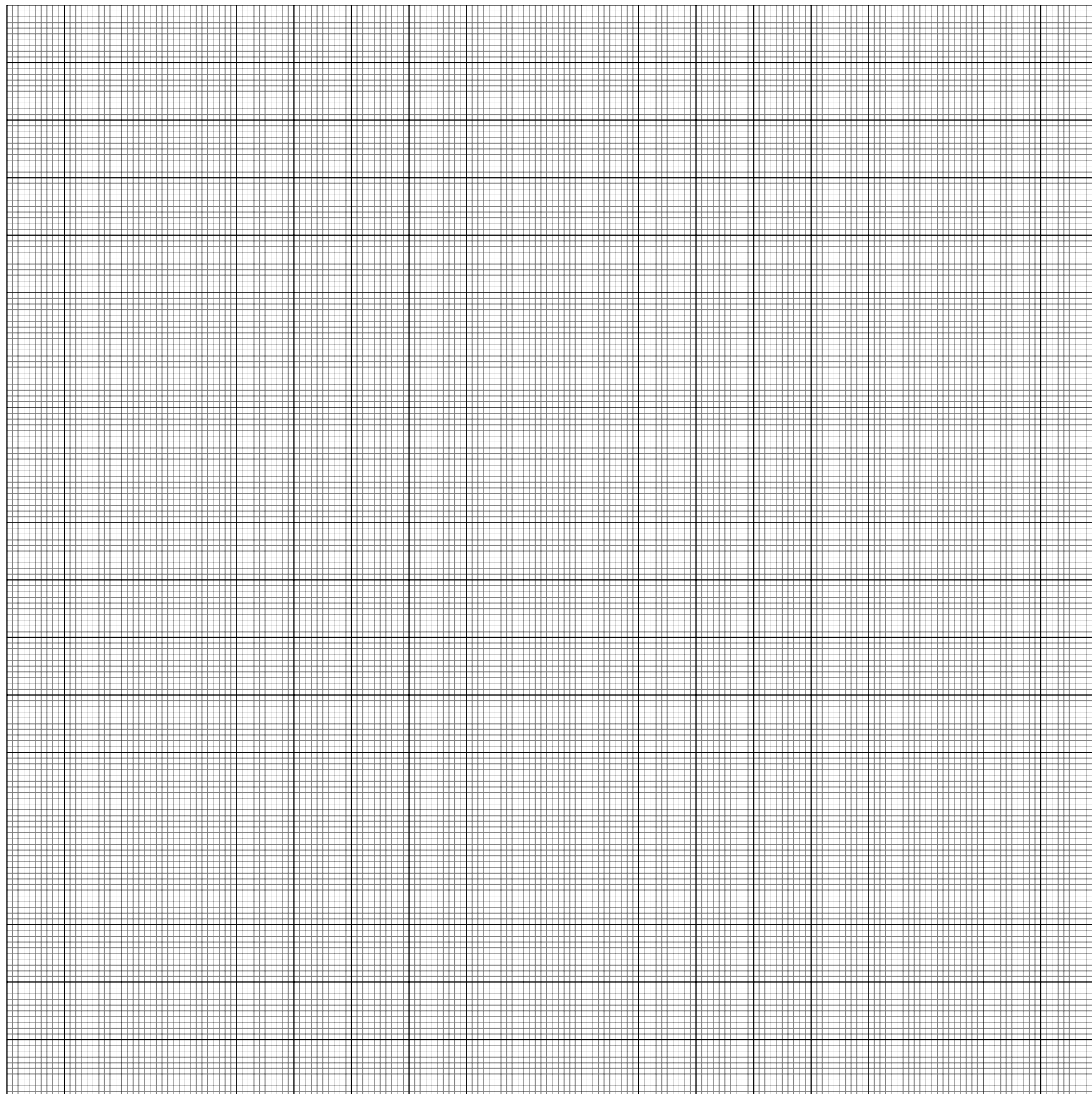
Mean molar mass: _____ g/mol
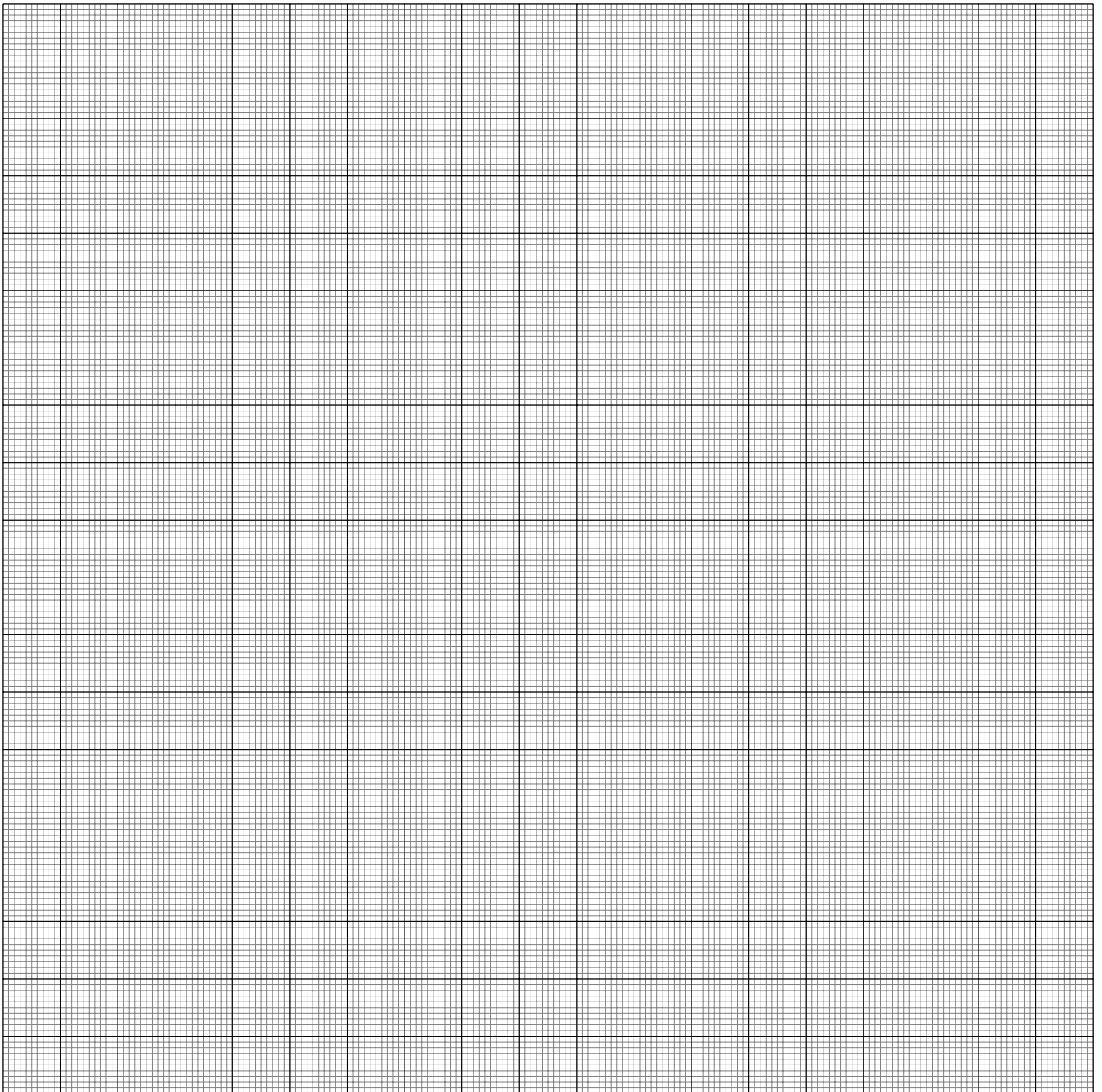
Figure 1: Temperature (Y axis) vs. time (X axis)

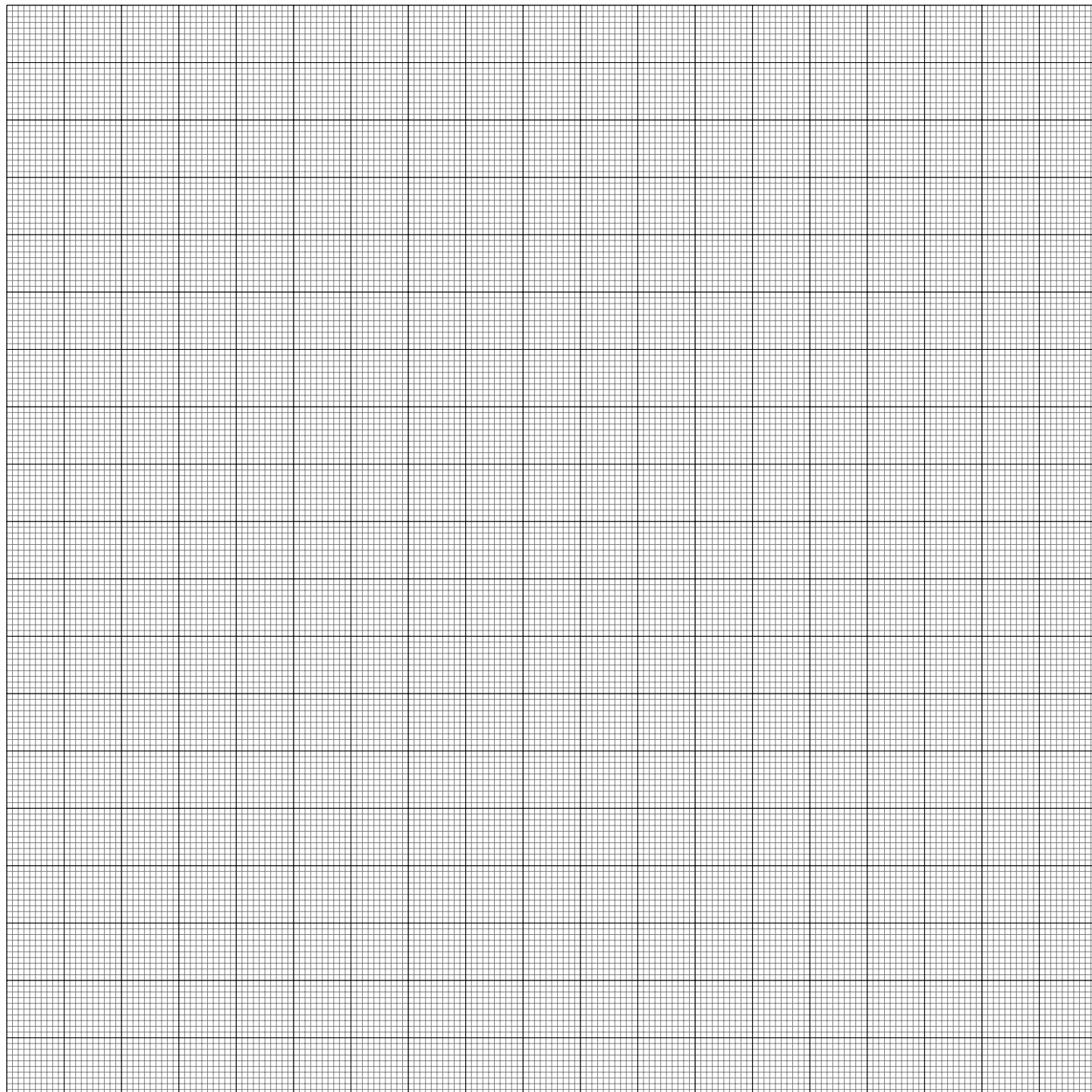Figure 2: Temperature (Y axis) vs. time (X axis)
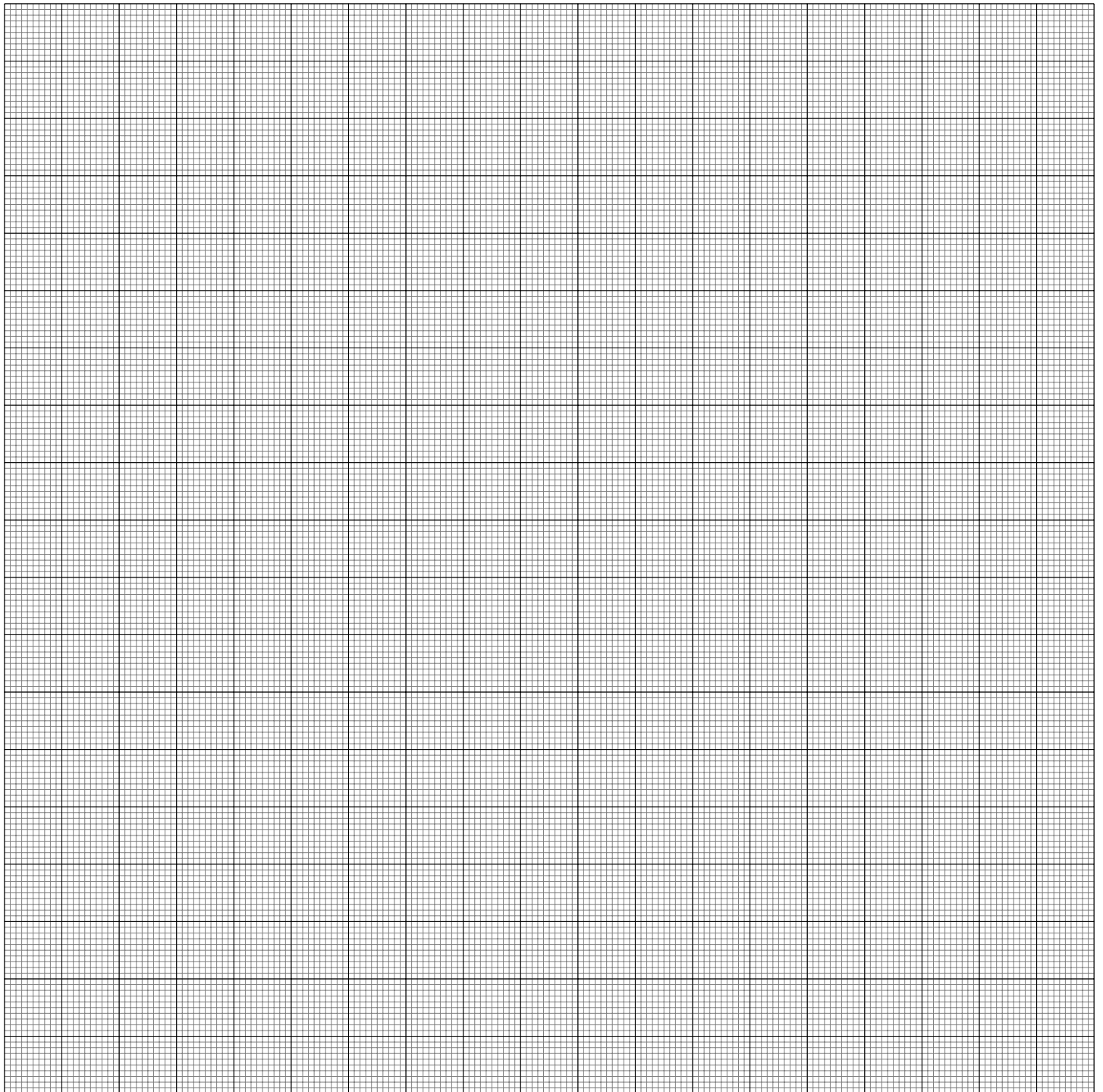
Figure 3: Temperature (Y axis) vs. time (X axis)

Figure 4: Temperature (Y axis) vs. time (X axis)

Post-lab Questions

Freezing point depression

1. If the empirical formula of the solute used in this experiment is C_3H_2Cl, what is the molecular formula?

2. What would be the effect on the molar mass in the following situations? Explain why.

    (a) A decrease in the mass measured for the solute.

    (b) An increase in the mass measured for the solute.

    (c) Dirt (soluble) from the stopper was added to the solution.

    (d) A loss of cyclohexane.

3. How much would the molar mass change if 1.000 g of the cyclohexane evaporates during part B? What would be the induced error?