

INTRODUCTION

The following experiment deals with the isolation process of an essential oil from seeds by means of hydro-distillation technique. Hydro-distillation is a method of distillation employing boiling water to extract essential oils from certain raw materials. Hydro-distillation is so far becoming the cheapest and most general distillation method employed in the isolation of essential oils from plant materials.

In practice, the raw material is soaked in water, then the mixture is heated to boiling and the distillates are collected after being cooled in the condenser. Due to polarity difference between isolated essential oils and water, the essential oils normally do not mix with water and accordingly separate from the water layer. Separation of the oils by means of an external separating funnel would result in the production of crude essential oils. Further purification technique is often required to obtain pure components present in the isolated essential oils.

The plant material used in this experiment is the fruit and the seed of *Myristica fragrans* Houtt, an evergreen tree, native of the East Moluccas, Indonesia. The seed of plant is known as ‘nutmeg’ and attached with arillus (specialized covering of a seed that partly or completely covered the seed), and is used for flavoring food and medicinal purposes. In this experiment, the participants will perform the following experiments related to “nutmeg and hydro-distillation”.

In this competition, you will perform the experimental procedure that will be used to answer all the questions in Physics, Biology and Chemistry. Read each step of the procedure thoroughly and carefully.

The hydro-distillation apparatus consists of:

1. Electric stove
2. Three-necks cylindrical boiling flask
3. Glass Allihn Condenser, Ball Shape Column
4. Modified Dean-Stark apparatus connected to Condenser equipped with water inlet and outlet
5. Rubber tubing (not shown)
6. Stand
7. Clamp holder
8. Clamp universal
9. Water bucket (not shown)
10. Aquarium pump (not shown)
11. Thermometers
12. 600 mL glass beaker (not shown)

The supporting equipment consists of:

- a. 10 mL volumetric (graduated) cylinder
- b. Plastic funnel
- c. Sample tube
- d. Cutting board
- e. Gloves
- f. Magnifying glass
- g. Goggles
- h. Rubber Stopper

Materials:

- a. Nutmeg fruit
- b. Nutmeg seed powder
- c. Water
- d. Boiling stone



Figure 1. Hydrodistillation Apparatus, will be used to isolate nutmeg oil from nutmeg seeds

Experimental Procedure

1. Make sure that the experimental equipment has been installed properly.
2. Fill the flask with 400 mL of water. Put the thermometer into the flask. Ensure that the tip of thermometer does not touch the bottom of the flask.
3. Turn on the electric stove (adjust the power of the electric stove to setting number 3) and at the same time push the stopwatch START button. **BE CAREFUL. DO NOT TOUCH THE ELECTRIC STOVE PLATE. The stove is equipped with automatic ON-OFF to maintain the temperature and to prevent overheating.**
4. Record the temperature of the water every 0.5 minute up to 12 minutes. Write the data on the answer sheet. After an interval of 12 minutes turn off the stove, and turn off the stopwatch.
5. Take the thermometer out and replace with cap provided.
6. With the water still in the flask, add more water up to 500 mL. Pour nutmeg powder that has been provided (120 grams) to the flask by using a funnel that has been provided. Add 3-5 pieces of boiling stone provided to the flask as well.
7. Turn on the electric stove, adjust the power to maximum setting (setting number 5). Continue heating until 90 minutes. Use stopwatch provided.
8. While waiting the 90 minutes, answer the **PART ONE: Physics, The effectiveness of energy absorption by water** questions on the answer sheet.
9. After you finish working on the problems of PART ONE, continue to work on **Biology experiments** as follows.
10. On the table you have been provided with a nutmeg fruit (including seed), the longitudinal and cross sections of nutmeg fruits and seeds.
11. Examine the nutmeg fruit and its parts carefully.
12. Examine the longitudinal and cross sections of fruit and seed and their parts.
13. Answer **PART TWO: Biology, Characteristics of Nutmeg** questions on the answer sheets provided.
14. After 90 minutes heating of the flask, turn off the electric stove. Measure the volume of nutmeg oil that has been produced in the Dean-stark. You may have to wait a while until the nutmeg oil is separated from water.

15. Transfer the entire collected nutmeg oil in the sample tube provided, close it, label it and leave it together with your answer sheets. Write your team code on the label provided and stick it to the sample tube.
16. Answer **PART THREE: Chemistry, Nutmeg Oil Distillation** questions on the answer sheets provided.

QUESTIONS

PART ONE: Physics, The effectiveness of energy absorption by water [13 point]

Transitions between solid, liquid, and gaseous phases typically involve large amounts of heat absorbed. If heat were added at a constant rate to a mass of ice to take it through its phase changes to liquid water and then to steam, the energies required to accomplish the phase changes (called the latent heat of fusion and latent heat of vaporization) would lead to plateaus in the temperature vs time graph. The graph below (Figure 1) presumes that the pressure is one standard atmosphere.

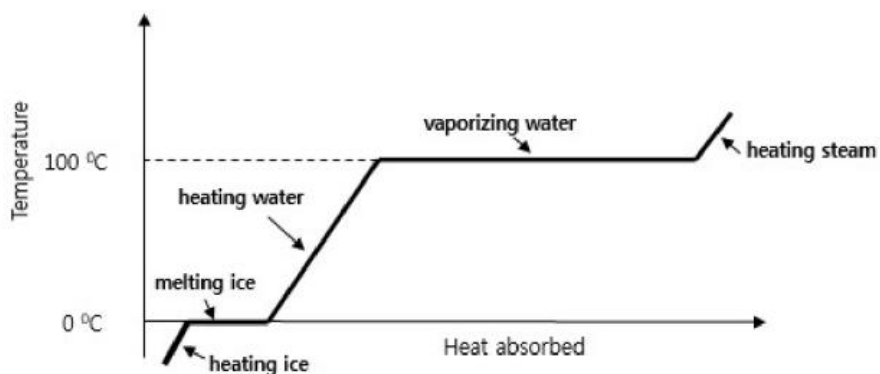


Figure 1. Graph of temperature versus heat absorbed

- Ph-1 [3.0 points]** Make a graph of the temperature ($^{\circ}\text{C}$) of water as a function of time (in minute).
- Ph-2 [1.5 points]** Determine the linear range of water temperature change (ΔT) and time change (Δt).
- Ph-3 [2.0 points]** Calculate the rate of change of temperature of water (in $^{\circ}\text{C}/\text{s}$) with respect to time by using the linear part of the graph (which means linear process in water).
- Ph-4 [2.0 points]** Calculate how much electrical energy (in Joule) is used within the linear part of the graph (electric power used by the stove is 600W).

- Ph-5 [1.5 points]** Calculate how much heat (in Joules) is used to increase the temperature of the water in the linear part of the graph. (Note that $c_{\text{water}} = 4180 \text{ J/kg}\cdot^{\circ}\text{C}$ and $\rho = 1000 \text{ kg/m}^3$).
- Ph-6 [1.5 points]** Calculate how much heat (in Joule) is released into the environment within the linear part of the graph.
- Ph-7 [1.5 points]** Calculate the percentage of energy used to raise the temperature of water with respect to the total energy of the stove within the linear part of the graph.

PART TWO: Biology, Characteristics of Nutmeg [13 points]

A. Nutmeg Fruit

Bi-1 [2.0 points] Draw the longitudinal section of the fruit with the seed intact.

Bi-2 [3.0 points] Label parts of the fruit with reference provided on the answers box.

Show the fruit parts by arrows. Choose the corresponding parts from the answers box and write down the answer by writing the letter only (for example A, B, C etc.).

B. Nutmeg Seed

Bi-3 [3.0 points] Draw the cross section of the seed.

Bi-4 [2.0 points] Label parts of the seed with reference provided on the answers box.

Show the seed parts by arrows. Choose the corresponding parts from the answers box and write down the answer by writing the letter only (for example A, B, C etc).

Answers Box

A. Receptacle	E. Locule	I. Seed	M. Embryo
B. Mesocarp	F. Nucellus	J. Testa/Seed Coat	N. Exocarp
C. Peduncle	G. Endosperm	K. Arillus	O. Placenta
D. Perisperm	H. Endocarp	L. Funiculus	P. Hypanthium

C. Nutmeg Fruit and Seed Characteristics

Fruit and Seed Classification:

Simple fruit	:	A fruit that develops from a single pistil
Compound fruit	:	A fruit in which one flower contains several separate ovaries which merge during development (aggregate) or a fruit in which several flowers, each with an ovary, develop into small fruits that are clustered or fused together into a larger fruit (multiple)
True fruit	:	A fruit in which all tissues are derived from a ripened ovary and its contents
Accessory fruit	:	A fruit that develops from a ripened ovary or

		ovaries but includes a significant portion derived from non-ovarian tissue
Fleshy fruit	:	A fruit that has a soft and pulpy wall at maturity
Dry fruit	:	A fruit that has a dry wall at maturity
Pome	:	A fruit that derived from several carpels, receptacle and outer portion
Drupe	:	A fruit that derived from a single carpel and containing (usually) one seed
Monocotyledon	:	Having a single cotyledon in the seed
Dicotyledon	:	Having two cotyledons in the seed
Round	:	Having the shape of a sphere or ball
Ovoid	:	Egg-shaped with the broader end at the base

Bi-5 [3.0 points]

Examine the fruit and the seed carefully. Tick (✓) one correct answer on each classification categories (A-F) in the box provided below.

Question:

Answer:

- | | | |
|-----------------------|--|--|
| A. Fruit origin: | <input type="checkbox"/> Simple fruit | <input type="checkbox"/> Compound fruit |
| B. Fruit composition: | <input type="checkbox"/> True fruit | <input type="checkbox"/> Accessory fruit |
| C. Fruit description: | <input type="checkbox"/> Fleshy fruit | <input type="checkbox"/> Dry fruit |
| D. Fruit type: | <input type="checkbox"/> Pome | <input type="checkbox"/> Drupe |
| E. Seed cotyledon: | <input type="checkbox"/> Monocotyledon | <input type="checkbox"/> Dicotyledon |
| F. Seed shape: | <input type="checkbox"/> Round | <input type="checkbox"/> Ovoid |

PART THREE: Chemistry, Nutmeg Oil Distillation [14.0 points]

After conducting experiment by using 120 g of ground nutmeg seed, you have obtained certain amount of nutmeg oil.

Ch-1 [4.50 points] How much is the volume of nutmeg oil you have obtained?

Ch-2 [1.50 point] It is known that the mass of exactly 1.00 mL of nutmeg oil is 0.862 g at 25 °C. What is the percentage by mass of nutmeg oil in nutmeg seed according to your experiment if it is measured at 25 °C?

Ch-3 [3.00 point] It is known that the main component of nutmeg oil is myristicin. Assume that your sample of nutmeg oil contains 65% of myristicin ($C_{11}H_{12}O_3$) by mass.

(a) [1.5 point] Calculate the number of myristicin molecules in your sample.

(b) [1.5 point] Calculate the mass of the carbon in grams in the myristicin in your sample. (atomic mass of C = 12, H = 1, and O = 16)

Ch-4 [1.00 point] Based on the result of your experiment, calculate how many kilograms of nutmeg seed powder are required to produce 100 grams of nutmeg oil?

Ch-5 [0.50 point] What is the function of boiling stones added in your experiment?

(a) to accelerate the heating of water

(b) to speed up the separation of nutmeg oil from water

(c) to assist the distribution of heat inside the cylindrical flask content.

Ch-6 [0.50 point] What is the main aim of using nutmeg seed powder rather than nutmeg seed granules in your experiment?

(a) to increase the solubility of nutmeg seed in water

(b) to increase the contact surface of nutmeg seed and water

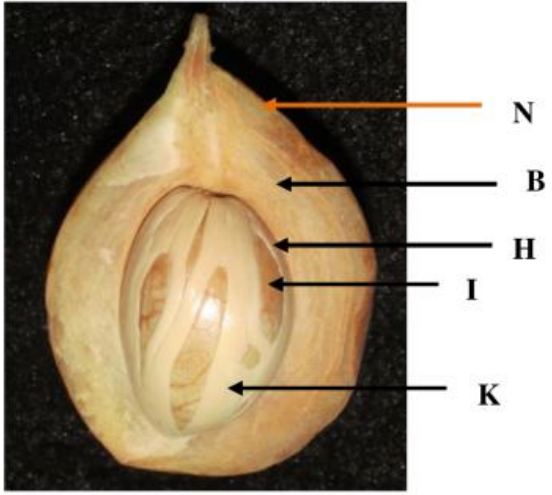
(c) to speed up the evaporation of water in the flask.

- Ch-7 [0.75 point]** The separation of water and nutmeg oil in the Dean-Stark apparatus reflects the principle of
- (a) like dissolves like
 - (b) vapor pressure difference
 - (c) chemical equilibrium.
- Ch-8 [0.75 point]** If the flow of cooling water in your experiment is changed from upper to lower part of the condensor, the condensation of the steam and nutmeg oil will be
- (a) more effective
 - (b) less effective
 - (c) no effect.
- Ch-9 [0.75 point]** Which of these following alternative separation techniques can be used to obtain nutmeg oil from the seed of nutmeg
- (a) Centrifugation
 - (b) Solvent extraction
 - (c) Paper chromatography
- Ch-10 [0.75 point]** What kind of changes in the experimental design would not reduce the yield of nutmeg oil
- (a) Heating too rapidly
 - (b) Using more boiling stones
 - (c) Using too short water condensor

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PART TWO: Biology, Characteristics of Nutmeg [13.0 points]

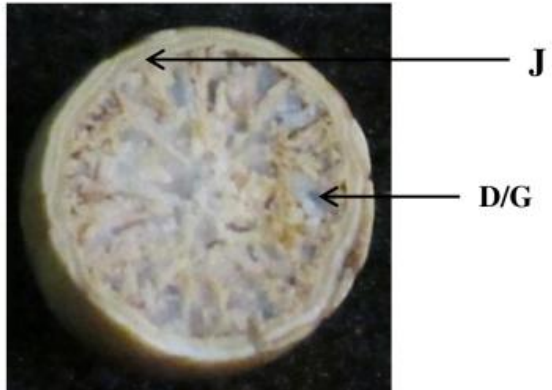
A. Nutmeg Fruit

Question	Bi-1. Draw the longitudinal section of the fruit with the seed intact! Bi-2. Label parts of the fruit with reference provided on the answers box. Show the fruit parts by arrows. Choose the corresponding parts from the answers box and write down the answer by writing the letter only (for example A, B, C etc.).	Points	Max
			
Bi-1	Drawing the correct longitudinal section of the fruit	0.25	2.0
	Drawing the cross section of the fruit	0.0	
	Size of the drawing is appropriate to the original fruit = 0.5 and correct proportion of the different parts = 0.5	1.0	
	Drawing the correct and complete 3 parts of the fruit (fruit, seed and arillus @ 0.25).	0.75	
	No drawing	0.0	
Bi-2	Correct labelling B = 0.6 point H = 0.6 point N = 0.6 point I = 0.6 point K = 0.6 point	3.0	3.0

Notes:

Pericarp	:	the walls of a ripen ovary or fruits (fruit coat: exocarp/epicarp, mesocarp and endocarp)
Seed	:	the fertilized mature ovule of flowering plant containing an embryo, the germ of propagative source, offspring or progeny)
Arillus/Aril/Mace	:	specialized outgrowth of a seed that partly or completely covered the seed

B. Nutmeg Seed

Question	Bi-3. Draw the cross section of the seed! Bi-4. Label parts of the seed with reference provided on the answers box. Show the seed parts by arrows. Choose the corresponding parts from the answers box and write down the answer by writing the letter only (for example A, B, C etc)	Points	Max
			
Bi-3	Drawing the correct cross section of the seed	0.5	3.0
	Drawing the longitudinal section of the seed	0.0	
	Size of the drawing is appropriate to the original seed	0.5	
	Correct drawing of the internal seed parts (testa/seed coat (J)= 1.0 point, endospem/perisperm (D/G))= 1.0 point Drawing the incomplete internal parts of the seed (without testa (J) or endospem/perisperm (D/G) = 1.0 point	2.0	
	No drawing	0.0	
Bi-4	Correct labelling : testa/seed coat (J)= 1.0 point, endospem/perisperm (D/G) = 1.0 point. Only one correct labeling: testa/seed coat (J) or endospem/perisperm (D/G) = 1.0 point	2.0	2.0
	No label	0.0	

Notes:

- Seed coat/Testa : the outer protective covering of a seed
 Endosperm : a tissue produced inside the seeds of most flowering plants it surrounds the embryo and provide nutrition in the form of starch, it can also contain oils and protein

C. Nutmeg Fruit and Seed Characteristics

Question (Points)	Fruit and Seed Classification Tick (✓) one correct answer on each classification categories (A-F) in the box provided below! (0.5 point for each correct answer)				
Bi-5 (3.0)	A. Fruit origin:	<input checked="" type="checkbox"/>	Simple fruit	<input type="checkbox"/>	Compound fruit
	B. Fruit composition:	<input checked="" type="checkbox"/>	True fruit	<input type="checkbox"/>	Accessory fruit
	C. Fruit description:	<input checked="" type="checkbox"/>	Fleshy fruit	<input type="checkbox"/>	Dry fruit
	D. Fruit type:	<input type="checkbox"/>	Pome	<input checked="" type="checkbox"/>	Drupe
	E. Seed cotyledon:	<input type="checkbox"/>	Monocotyledon	<input checked="" type="checkbox"/>	Dicotyledon
	F. Seed shape:	<input type="checkbox"/>	Round	<input checked="" type="checkbox"/>	Ovoid

PART THREE: Chemistry, Nutmeg Oil Distillation [14.0 points]

After conducting experiment by using 120 g of ground nutmeg seed, you have obtained certain amount of nutmeg oil.			
Question	How much is the volume of nutmeg oil you have obtained?	Points	Max
Ch-1	Sample collected by students is free of water	2.00	4.50
	Volume of Nutmeg Oil (mL):		
	> 4.00	2.50	
	2.50 – 3.99	2.00	
	1.00 – 2.49	1.50	
	0.00 – 0.99	0.50	
	If wrong fraction (only water) is collected	0.00	
Question	It is known that the mass of exactly 1.00 mL of nutmeg oil is 0.862 g at 25 °C. What is the percentage by mass of nutmeg oil in nutmeg seed according to your experiment if it is measured at 25 °C?	Points	Max
Ch-2	Mass of nutmeg oil = volume (mL) x density (g/mL) =..... mL x 0.862 (g/mL) = g	0.75	1.50
	Percentage of nutmeg oil in nutmeg seed: = {mass of nutmeg oil (g)/mass of nutmeg seed (g)} x 100 % = %	0.75	
Question	It is known that the main component of nutmeg oil is myristicin. Assume that your sample of nutmeg oil contains 65% of myristicin (C ₁₁ H ₁₂ O ₃) by mass. (a) [1.5 point] Calculate the number of myristicin molecules in your sample. (b) [1.5 point] Calculate the mass of the carbon in grams in the myristicin in your sample. (atomic mass of C = 12, H = 1, and O = 16)	Points	Max
Ch-3	Molecular mass of myristicin = (12 x 11) + (1 x 12) + (16 x 3) = 192	0.50	3.00
	Mass of myristicin in nutmeg oil: = volume of nutmeg oil (mL) x density (g/mL) x 0.65 = x 0.862 x 0.65 g	0.50	
	Number of moles of myristicin: = (gram of myristicin)/192 = moles	0.50	
	Number of molecules of myristicin: = number of mole of myristicin x Avogadro number = number of mole of myristicin x 6.02x10 ²³ molecule = x10 ²³ molecules	0.50	

	Mass of carbon in the myristicin of your nutmeg oil: = $132/192 \times$ gram of myristicin = grams	1.00	
Question	Based on the result of your experiment, calculate how many kilograms of nutmeg seed powder are required to produce 100 grams of nutmeg oil?	Points	Max
Ch-4	Assume that the percentage of nutmeg oil in nutmeg seed obtained from question Ch-3 = a % The mass of nutmeg seed powder required to produce 100 grams (0.1 kg) of nutmeg oil: = $(0.1 \times 100)/a$ kg =kg	1.00	1.00
Question	What is the function of boiling stones added in your experiment? (a) to accelerate the heating of water (b) to speed up the separation of nutmeg oil from water (c) to assist the distribution of heat inside the cylindrical flask content.	Points	Max
Ch-5	Answer: (c) to assist the distribution of heat inside the cylindrical flask content.	0.50	0.50
Question	What is the main aim of using nutmeg seed powder rather than nutmeg seed granules in your experiment? (a) to increase the solubility of nutmeg seed in water (b) to increase the contact surface of nutmeg seed and water (c) to speed up the evaporation of water in the flask.	Points	Max
Ch-6	Answer: (b) to increase the contact surface of nutmeg seed and water	0.50	0.50
Question	The separation of water and nutmeg oil in the Dean-Stark apparatus reflects the principle of (a) like dissolves like (b) vapor pressure difference (c) chemical equilibrium.	Points	Max
Ch-7	Answer: (a) like dissolves like	0.75	0.75
Question	If the flow of cooling water in your experiment is changed from upper to lower part of the condenser, the condensation of the steam and nutmeg oil will be (a) more effective (b) less effective (c) no effect.	Points	Max
Ch-8	Answer (b) less effective	0.75	0.75

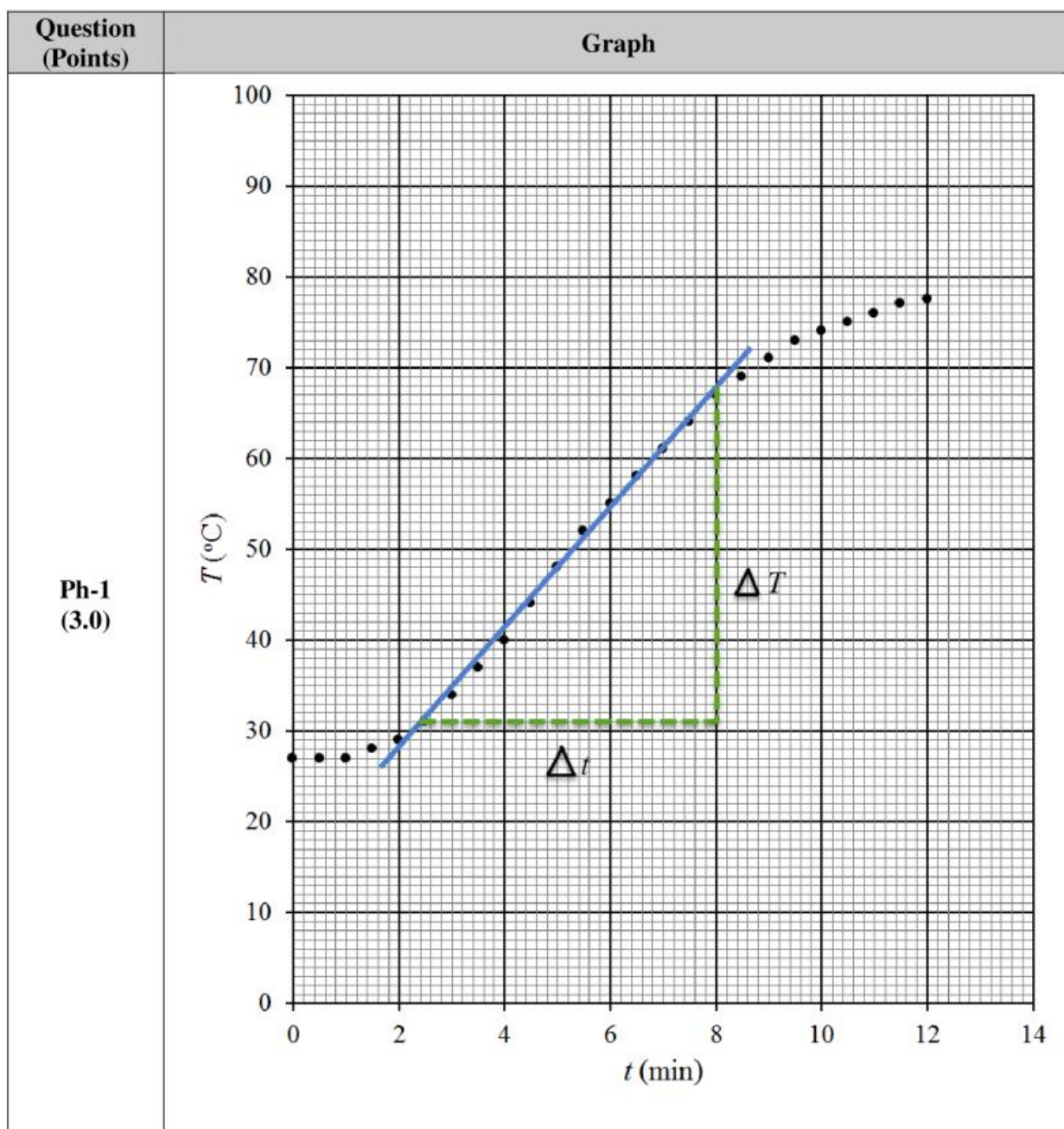
Question	Which of these following alternative separation techniques can be used to obtain nutmeg oil from the seed of nutmeg (a) Centrifugation (b) Solvent extraction (c) Paper chromatography	Points	Max
Ch-9	Answer: (b) Solvent extraction	0.75	0.75
Question	What kind of changes in the experimental design would not reduce the yield of nutmeg oil (a) Heating too rapidly (b) Using more boiling stones (c) Using too short water condensor	Points	Max
Ch-10	Answer: (b) Using more boiling stones	0.75	0.75

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PART ONE: Physics, The effectiveness of energy absorption by water [13.0 points]

Experimental Data

<i>t</i> (min)	0.0	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0
<i>T</i> (°C)	27	27	27	28	29	31	34	37	40
<i>t</i> (min)	4.5	5.0	5.5	6.0	6.5	7.0	7.5	8.0	8.5
<i>T</i> (°C)	44	48	52	55	58	61	64	67	69
<i>t</i> (min)	9.0	9.5	10.0	10.5	11.0	11.5	12.0		
<i>T</i> (°C)	71	73	74	75	76	77	77.5		



Question	Make a graph of the temperature of water as a function of time!	Points	Max
Ph-1	Name of both axis	0.5	3.0
	Unit of axis	0.5	
	Accuracy point positions of data (correctness of the data plotting)	2.0	
Question	Determine the linear range of water temperature change (ΔT) and time change (Δt)!	Points	Max
Ph-2 (1.5)	ΔT in the linear part	0.75	1.5
	Δt in the linear part	0.75	
	No answer or any other value	0.0	0.0
Question	Calculate the rate of water temperature change (in $^{\circ}\text{C}/\text{s}$) with respect to time by using the linear part of the graph(which means linear process in water)!	Points	Max
Ph-3 (2.0)	Slope = (depending on the linear part of the graph)		2
	No answer or any other value		0.0
Question	Calculate how much electrical energy (in joule) is used within the linear part of the graph (electric power used by the stove is 600W)!	Points	Max
Ph-4 (2.0)	Electric energy = $E_E = P \times \Delta t$		
	$E_E = (600\text{W}) \times (\Delta t \text{ [in minute]}) \times (60\text{s})$, (Δt depends on student's linear part of graph) Correct formula = 1.0 point	1	2.0
	Correct calculation (in joule) = 1.0 point (units may not be stated)	1	
	$E_E = (600\text{W}) \times (\Delta t \text{ [in minute]})$, (Δt depends on student's linear part of graph) Correct calculation	0.5	1.0
	Correct formula	0.5	
	(units may not be stated)		

Question	Calculate how much heat (in joule) is received by water within the linear part of the graph! (Note that $c_{water} = 4180 \text{ J/kg}\cdot^{\circ}\text{C}$).	Points	Max
Ph-5 (1.5)	$\rho_{water} = 1 \text{ g/cm}^3$ $c_{water} = 4180 \text{ J/kg}\cdot^{\circ}\text{C}$ $V_{water} = 400 \text{ mL}$ $m_{water} = \rho_{water} \times V_{water} = (1) \times (400) = 400 \text{ g} = 0.4 \text{ kg}$ $Q_{water} = m_{water} \times c_{water} \times \Delta T$		
	Correct formula	0.5	1.5
	Correct calculation (in joule)	1.0	
	Correct calculation (units may not be stated)	0.5	1.0
	Correct formula	0.5	
	No answer or any other value	0.0	0.0
Question	Calculate how much heat (in joule) is released into the environment during the linear part of the graph!	Points	Max
Ph-6 (1.5)	$\Delta Q = E_E - Q_{water}$		
	Correct formula	0.75	1.5
	Correct calculation (in joule)	0.75	
	Correct calculation (units may not be stated)	0.5	1.0
	Correct formula	0.5	
	No answer or any other value	0.0	0.0

Question	Calculate the percentage of energy absorbed by water with respect to the total energy of the stove within the linear part of the graph!	Points	Max
Ph-7 (1.5)	$\eta = (mc\Delta T) \div (P\Delta t) \times 100\%$		
	Correct formula	0.5	1.5
	Correct calculation (in joule)	1.0	
	Correct calculation (units may not be stated)	0.5	1
	Correct formula	0.5	
	No answer or any other value	0.0	0.0