

# CHEM 350 Prelab Questions

Welcome to the CHEM350 Online Prelab Questions!

These questions are all 'Open Book' and do not count for any marks in the lab component. They are here to help you prepare and understand what you need to know before you attend your supervised labs.

You will need to read the experiments in the Laboratory Manual (Full).

We estimate it should take you 10 minutes in which to complete each set of prelab questions.

## Experiment 1 – Melting-point Determinations

1. Why do we need to know the melting point of a substance?
  - a. To determine the exact time it takes for a sample to melt, and what colour the compound becomes after heating.
  - b. To determine the purity of a sample, 1-2 °C range = pure, 3 °C range or more = impure.
  - c. To identify and then determine the crystal lattice structure of a compound.
  - d. To identify the compound before using it in a reaction.
2. List the three steps used to prepare a melting point sample?
  - a. (1) Mix the solid well before sampling. (2) Transfer the compound to a melting point tube. (3) Pack the sample to a height ~1 mm.
  - b. (1) Transfer a small amount of compound to a melting point tube. (2) Pack the sample to a height ~5 mm. (3) Place the packed sample into the melting point apparatus and begin heating.
  - c. (1) Crush the solid using a mortar and pestle. (2) Transfer a small amount of powder to a melting point tube. (3) Pack the sample to a height ~1 mm.
  - d. (1) Crush the solid in a mortar. (2) Transfer a large amount of powder to a melting point tube. (3) Only pack the sample if the melting point tube is too full.
3. How does the melting point of an impure compound generally differ from a pure compound?
  - a. higher melting point and smaller range.
  - b. lower melting point and smaller range.
  - c. higher melting point and larger range.
  - d. lower melting point and larger range.
4. Define the temperatures recorded at the beginning and end of the melting point range.
  - a. the melting points happen slow and are very hard to see.
  - b. lower limit = when the first drop of liquid is seen; upper limit = when the sample is completely liquid.
  - c. lower limit = when the sample is completely liquid; upper limit = when the sample has evaporated from the melting point tube.
  - d. lower limit = when the sample begins to shrink/shrivel; upper limit = when the sample is completely liquid.

5. The CRC Handbook of Chemistry and Physics sometimes reports the melting point of a compound as a single number. What does this mean?
- It's the midpoint value between the upper and lower limit of the melting point range.
  - It's the lower limit of the melting point range.
  - It's the upper limit of the melting point range.
  - It's the upper limit for the melting point range, corrected for barometric pressure effects.
6. The melting point apparatus should be heating at what ramp rate (? C/min) as it approaches the melting point of the compound?
- The maximum rate of heating. Use the boost switch.
  - 3
  - 2
  - 1
7. What is the name of the piece of laboratory equipment on the right?
- melting point apparatus
  - pan balance
  - spectrophotometer
  - pH meter



8. The identification of a unknown solid will be achieved by mixed melting point determination.
- True
  - False

## Experiment 1 - Lab Safety

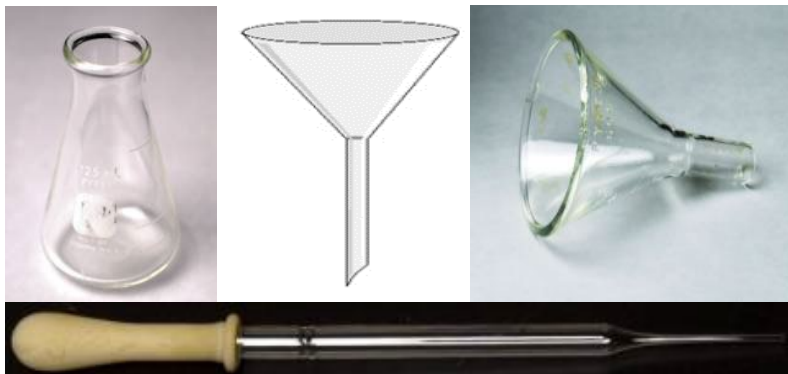
1. 'WHMIS' stand for?
  - a. Workplace Hazardous Materials Information and Symbols
  - b. Worker Hazardous Materials Information System
  - c. Workplace Hazardous Materials Information System
  - d. Worker Hazardous Materials Information Sheets
  
2. 'MSDS' stand for?
  - a. Most Severe Data Sheet
  - b. Material Sheet for Dangerous Supplies
  - c. Material Safety Danger Sheet
  - d. Material Safety Data Sheet

## Experiment 2 – Recrystallization

1. Why does a chemist recrystallize an organic compound?
  - a. To determine the solubility of the compound in a particular solvent.
  - b. To convert the compound into a eutectic mixture.
  - c. To identify the compound.
  - d. To purify a compound prior to analysis or use in a reaction.
  
2. Which statement briefly explains how recrystallization increases the purity of a compound?
  - a. Recrystallization is an art and it is by luck you get any pure crystals at all.
  - b. Assuming that impurities are highly soluble in the selected solvent at all temperatures, by dissolving the desired impure compound in a minimum of hot solvent, and then cooling the solution, brand new crystals form while the impurities stay in solution, resulting in purer than original crystals.
  - c. By dissolving the compound in hot solvent and then cooling the solution, brand new crystals form which are purer than the original crystals.
  - d. Assuming that impurities are only highly soluble in the hot solvent, by dissolving the desired impure compound in a minimum of hot solvent, and then cooling the solution, brand new crystals form while the impurities stay in solution, resulting in purer than original crystals.
  
3. The following are the 5 steps of the recrystallization procedure:
  - i. Select the recrystallization solvent.
  - ii. Dissolve the crude solid in a minimum amount of hot solvent (= saturated solution).
  - iii. Make a decision to hot gravity filter or not.
  - iv. Slow cool and allow the crystals to form.
  - v. Collect the crystals using vacuum filtration and wash with ice cold recrystallization solvent, and then allow the crystals to dry to a constant weight.
  - a. True
  - b. False

4. What are the criteria for selecting a solvent suitable for a single solvent recrystallization?
  - a. soluble in hot solvent and soluble in cold solvent.
  - b. insoluble in hot solvent and insoluble in cold solvent.
  - c. insoluble in hot solvent and soluble in cold solvent.
  - d. soluble in hot solvent and insoluble in cold solvent.
  
5. Boiling stones must be added to the recrystallization solvent prior to heating. Why (Note: there are two very good reasons for doing so)?
  - a. In order to prevent the solution from 'bumping' in the first place.
  - b. To avoid a sudden violent eruption of liquid should the solution be hot when the stones are added.
  - c. a and b are correct.
  - d. none of the above.
  
6. What are two situations where you are required to perform a hot gravity filtration.
  - a. colored impurities are present (therefore charcoal was added), and to remove soluble impurities.
  - b. soluble impurities are present (therefore charcoal was added), and to remove insoluble impurities.
  - c. colored impurities are present (therefore charcoal was added), and to remove insoluble impurities.
  - d. to remove boiling stones that were added, and to remove soluble impurities.

7. Which piece of equipment is not normally used to perform a recrystallization:



- a. Erlenmeyer flask  
b. Long narrow stemmed glass funnel  
c. Short stemmed wide mouthed glass funnel  
d. Pasteur pipette
8. If too much solvent is used when making a saturated solution, the yield of crystals at the end of the recrystallization will be reduced.
- a. True  
b. False

## Experiment 2 - Lab Safety

1. Which of the four following statements is false?
- a. Never heat a flammable solvent directly on a hot plate.  
b. Never heat an Erlenmeyer flask filled more than 2/3rds full of liquid.  
c. Always remember to use boiling stones when heating a solvent.  
d. Acetanilide waste can be washed down the sink drain.
2. Acetanilide waste should be placed into the
- a. General Organic Waste bottle in the fumehood.  
b. Halogenated Organic Waste bottle in the fumehood.  
c. Flushed down the drain with water.

## Experiment 3 – Distillation: Simple and Fractional

1. Why does a chemist distill an organic liquid?
  - a. To determine the polarity of a particular solvent.
  - b. To purify a compound prior to analysis or use in a reaction.
  - c. To identify the compound.
  - d. To convert the compound into its vapour form.
  
2. Which statement best explains how distillation purifies a compound?
  - a. Distillation is used to concentrate a desired compound by removing undesired impurity vapours.
  - b. Assuming that the impurity has a higher boiling point (must be greater than 25-30° C different than the solvent), the desired solvent can be separated by heating it to its vapour phase and then collecting the pure vapours in a receiver flask.
  - c. Distillation involves heating a liquid to its boiling point, at atmospheric or reduced pressure, to convert it to its vapour, and then condensing the vapour back into a liquid by cooling with a condenser and collecting the condensate in a receiving flask.
  - d. Distillation involves heating a liquid to its boiling point to convert it to its vapour, and then converting the vapour back to liquid by icing the receiver flask.
  
3. Perfect soft-boiled eggs take 4:48 minutes to cook in Vancouver. To obtain the same result in Banff they need 9:20 minutes in boiling water. Why?
  - a. Banff tends to be colder than Vancouver.
  - b. It is far easier to boil water in Vancouver than in Banff.
  - c. It takes less heat at higher altitudes to get water to boil.
  - d. The eggs available in Banff are more raw and require longer cooking times.
  
4. Boiling stones must be added to the distillation flask prior to heating. Why?
  - a. In order to prevent the solution from 'bumping'.
  - b. To avoid a sudden violent eruption of liquid should the solution already be hot when the stones are added.
  - c. To promote smooth boiling of the solvent.
  - d. all of the above.



5. Which is the name of the piece of glassware on the right?
- Claisen adaptor
  - three-way connector or still head
  - vacuum take-off adaptor
  - thermometer adaptor



6. Simple distillations can be performed on a mixture of two solvents only if the boiling points are different by more than 15-20° C. Fractional distillations are performed on a mixture of two solvents if the boiling points are different by less than 25-30° C.
- True
  - False

### Experiment 3 Lab Safety

1. Which of the four following statements is false?
- Never heat a distillation flask to dryness.
  - Never heat a round bottom flask filled more than 1/2 full of liquid.
  - Always remember to use boiling stones when heating a solvent.
  - Ensure you have an open system before you begin heating.
  - Once your distillation apparatus is fully assembled, then insert your thermometer down through the thermometer adaptor.
2. When setting up the condenser, adjust the cooling water flow so that:
- it flows in the bottom and out the top of the condenser, at a maximum flow rate.
  - it flows in the top and out the bottom of the condenser, at a maximum flow rate.
  - it flows in the bottom and out the top of the condenser, at a minimum flow rate.
  - it flows in the top and out the bottom of the condenser, at a minimum flow rate.

## Experiment 4 – Refractive Index

1. The two common ways to assess the purity of a liquid organic sample are:
  - a. thin layer chromatography, and determine its refractive index.
  - b. distill the liquid and determine its density by determining the mass and volume of product.
  - c. convert the compound into its vapour form and condense it back to a liquid.
  - d. fractional distillation of the liquid, followed by refractive index analysis.
2. The refractive index of a liquid is fundamentally based on the change of the speed of
  - a. flowing water.
  - b. gaseous molecules.
  - c. light.
3. The refractive index is dependent upon which two key factors?
  - a. temperature of the sample and the wavelength of the incident light.
  - b. the operator of the refractometer and the amount of sample used.
  - c. density of the sample and the wavelength of the incident light.
4. Which of the following sequences describes the correct order of the steps needed to measure a refractive index?
  - a. Turn on refractometer, apply sample, adjust side hand wheel, adjust thumb wheel for chromatic aberration, readjust side hand wheel, read meter.
  - b. Turn on refractometer, apply sample, adjust thumb wheel for chromatic aberration, adjust side hand wheel, readjust side hand wheel, read meter.
  - c. Turn on refractometer, adjust thumb wheel for chromatic aberration, adjust side hand wheel, apply sample, read meter
  - d. All of the above (the order does not matter).

5. From the formulae provided below, choose the one which describes the correct method to calculate the percentage error in a refractive index measurement.

a)  $= \frac{|\text{actual value} - \text{theoretical value}|}{\text{theoretical value}} \times 100\% =$

b)  $= \frac{\text{actual value}}{\text{theoretical value}} \times 100\% =$

c)  $= \frac{|\text{theoretical value} - \text{actual value}|}{\text{actual value}} \times 100\% =$

#### Experiment 4 Lab Safety

1. What is the major safety concern regarding this experiment?
- a) Handling of flammable and toxic solvents.
  - b) the sodium lamp in the refractometer.
  - c) keeping the refractometer from moving.
  - d) the temperature of the sample during refractometer readings.

## Experiment 5 – Extraction, separation and the use of drying agents

1. What is the easiest way to separate two immiscible liquids?
  - a. use a ultracentrifuge.
  - b. use a Büchner funnel.
  - c. use a separatory funnel.
2. Fifty milliliters of 5% sodium hydroxide (aq) and pure dichloromethane were added to a separatory funnel. What would you observe?
  - a. a homogeneous, clear, and colourless solution.
  - b. two layers of liquid, both clear and colourless.
  - c. two layers of liquid, fizzing, and the separatory funnel would have to be immediately vented.

3. Given  $K = ([\text{solute}] \text{ in solvent A, g L}^{-1}) / ([\text{solute}] \text{ in solvent B, g L}^{-1})$

The distribution coefficient for a compound in a two solvent extraction system is 2.0. If you are given 4.0 g of compound dissolved in 100 mL of solvent B, is the following the correct answer for how much compound will be extracted, if you use 50 mL of solvent A for the extraction.

$$K = 2.0 = \frac{(x / 0.05L)}{(4 - x) / 0.1L}, \text{ rearrange to solve for } x, = \frac{(8 - 2x)}{0.1L} = \frac{x}{0.05L} \text{ or } 0.1x = 0.05(8 - 2x), \text{ therefore, } 0.2x = 0.4 \text{ or } x = 2\text{g}$$

- a. Yes
  - b. No
4. Why do we add 5% NaOH to extract the organic acid from the organic mixture?
    - a. The strong, aqueous, inorganic base (NaOH) will react with the organic acid and convert the organic acid to its 'water insoluble', salt form (R-COO<sup>-</sup>Na<sup>+</sup>).
    - b. The strong, aqueous, inorganic base (NaOH) will react with the organic acid and convert the organic acid to its 'water soluble', salt form (R-COO<sup>-</sup>Na<sup>+</sup>).
    - c. The weak, aqueous, inorganic base (NaOH) will not react with the organic acid and not convert the organic acid to its 'water soluble', salt form (R-COOH + NaOH → no reaction).

5. Why do we add 1.5 M HCl to extract the organic base from the organic mixture?
  - a. The strong, aqueous, inorganic acid (HCl) will react with the organic base and convert the organic base to its 'water insoluble', salt form (R-NH<sub>3</sub><sup>+</sup>Cl<sup>-</sup>).
  - b. The strong, aqueous, inorganic acid (HCl) will react with the organic base and convert the organic base to its 'water soluble', salt form (R-NH<sub>3</sub><sup>+</sup>Cl<sup>-</sup>).
  - c. The weak, aqueous, inorganic acid (HCl) will not react with the organic base and not convert the organic base to its 'water soluble', salt form (R-NH<sub>2</sub> + HCl → no reaction).
  
6. Why do we add concentrated hydrochloric acid (6 or 12 M) to the separated, aqueous salt of the organic acid (R-COO<sup>-</sup>Na<sup>+</sup>(aq) + HCl(aq) → ?).
  - a. The strong, concentrated, aqueous, inorganic acid (6 or 12 M HCl) will react with the salt of the organic acid, and reconvert it to its 'water soluble', salt form (R-COO<sup>-</sup> H<sup>+</sup>).
  - b. The strong, concentrated, aqueous, inorganic acid (6 or 12 M HCl) will lower the pH of the organic salt solution, but will not react with the organic acid salt.
  - c. The strong, concentrated, aqueous, inorganic acid (6 or 12 M HCl) will react with the salt of the organic acid, and reconvert it to its 'water insoluble' form (R-COOH).
  
7. Why do we add concentrated sodium hydroxide (6 M) to the separated, aqueous salt of the organic base (R-NH<sub>3</sub><sup>+</sup>Cl<sup>-</sup>).
  - a. The strong, concentrated, aqueous, inorganic base (6 M NaOH) will react with the salt of the organic base, and reconvert it to its 'water soluble', salt form (R-NH<sub>2</sub><sup>+</sup> Cl<sup>-</sup>).
  - b. The strong, concentrated, aqueous, inorganic base (6 M NaOH) will raise the pH of the organic salt solution, but will not react with the organic base salt.
  - c. The strong, concentrated, aqueous, inorganic base (6 M NaOH) will react with the salt of the organic base, and reconvert it to its 'water insoluble' form (R-NH<sub>2</sub>).

## Experiment 5 Lab Safety

1. What is/are the safety concern(s) regarding this experiment?
  - a. A leaking separatory funnel.
  - b. dichloromethane is toxic and readily absorbed through the skin.
  - c. hot glassware and flammable solvents being used during the recrystallizations.
  - d. all the above.

## Experiment 6 – Infrared Spectroscopy Tutorial

There are no prelab questions as this is an exercise to be completed at home. An optional tutorial will be provided at the in-person lab by the instructor(s).

## Experiment 7 – Extraction of Usnic Acid from Lichen

- Which of the following compounds is optically active?
  - ultra pure water.
  - acetone.
  - tetrahydrofuran.
  - dichloromethane.
  - none of the above.
- The measured optical activity of a solid compound is affected by three major factors. They are:
  - concentration and temperature of the solution, and length of sample tube.
  - size of the molecule, natural source of chemical, and solubility.
  - density and temperature of compound, and length of sample tube.
- If 0.8000 g compound was dissolved in 50.00 mL of solvent, and the solution was placed in a 2 dm long sample tube, and gave an (observed rotation) of +3.2 degrees, the specific rotation would be:
  - +50°
  - +100°
  - +1000°
- During the solid-liquid extraction of the lichen with acetone, the lab manual (Exp. 7 Procedure Part A of lab manual) suggests we extract for 30 minutes. What would happen if the extraction went longer than 30 minutes?
  - The usnic acid being leached out of the lichen would begin to denature.
  - Nothing. A maximum amount of usnic acid has been extracted and further extraction time does no harm.
  - The extraction solvent, acetone, will begin to evaporate and thereby diminish the overall yield of usnic acid.
  - The experiment will have to be terminated because there will now be insufficient time to complete it.



5. Why is all the 'acetone extraction solvent' removed prior to beginning the recrystallization part of the procedure?
  - a. So that a saturated solution can be made using reagent grade acetone.
  - b. The acetone must be removed in order to isolate and characterize the crude usnic acid.
  - c. Acetone is not a suitable recrystallization solvent.
  - d. Tetrahydrofuran and acetone are miscible solvents so acetone must be removed prior to using the polarimeter.

### Experiment 7 Lab Safety

1. Which reagent(s) used in this experiment must be specially handled, and why?
  - a. acetone, as it is highly flammable.
  - b. tetrahydrofuran, as it is highly toxic and flammable.
  - c. a and b are correct.

## Experiment 8 – Preparation of Cyclohexene from Cyclohexanol

1. The preparing of cyclohexene from cyclohexanol is an example of a widely used method of converting an alcohol functional group into an functional group?
  - a. alkene
  - b. alkane
  - c. non-reactive.
  - d. reactive.
2. The purpose of boiling stones to the round bottom flask reaction vessel:
  - a. to promote bumping.
  - b. to promote smooth boiling.
  - c. to preserve the product
3. The purpose of adding phosphoric acid to the reaction vessel containing cyclohexanol is:
  - a. to neutralize any contaminating base.
  - b. to act as a catalyst in the reaction.
  - c. to slow the reaction rate and thereby increase the yield.
4. How do you separate the aqueous and the cyclohexene organic layer?
  - a. distillation.
  - b. reflux.
  - c. using a separatory funnel.
  - d. extraction.
5. The purpose of adding saturated sodium chloride (brine) to the aqueous layer in Step 6 of the procedure is to:
  - a. to make a salt of the organic acid.
  - b. to make the product less soluble in the water and to 'salt out' the water from the organic layer.
  - c. to preserve the product.
  - d. to add water to the organic layer.

6. Which of the following ways would characterize your final product and thereby help prove that you have converted cyclohexanol to cyclohexene:
- infrared spectroscopy.
  - nuclear magnetic resonance spectroscopy.
  - refractive index.
  - density.
  - all of the above.
  - only a and b are correct.
7. What is the first step called in the mechanism for an acid catalyzed dehydration?
- protonation.
  - elimination.
  - carbocation intermediate formation.
  - substitution.
8. Alexander Zaitzev's rule for elimination reactions states:
- "in the addition of HX to an alkene, the more highly substituted carbocation is formed as the intermediate rather than the less highly substituted one".
  - "Base-induced elimination reactions generally give the more highly substituted (more stable) alkene product".
  - "The structure of a transition state resembles the structure of the nearest stable species. Exergonic reaction steps resemble reactants and Endergonic reaction steps resemble products".

### Experiment 8 Lab Safety

1. Which reagent(s) used in this experiment must be specially handled, and why?
- saturated sodium chloride, as it is highly corrosive.
  - cyclohexanol, as it is a toxic starting reagent.
  - phosphoric acid, as it is highly corrosive.
  - both b and c.

## Experiment 9 – The Nitration of Acetanilide

1. What is the purpose of dissolving the acetanilide in glacial acetic acid prior to beginning the nitration reaction?
  - a. So that the acetanilide is 'in solution' when the sulfuric acid is added.
  - b. So that the acetanilide is 'in solution' when the nitrating mixture is added.
  - c. To stabilize the acetanilide prior to the addition of sulfuric acid.
  - d. To prevent the acetanilide from reacting too quickly when the nitrating mixture is added.
2. What happens when you mix sulfuric acid with nitric acid?
  - a. The sulfuric acid is the weaker acid so nitric acid converts it to sulfate ion.
  - b. Nitronium ion is formed.
  - c. a slow endothermic reaction occurs.
3. What is the name of the electrophile used in this experiment?
  - a. sulfuric acid.
  - b. nitric acid.
  - c. acetanilide.
  - d. nitronium ion.
4. What acts as the nucleophile in this experiment?
  - a. sulfuric acid.
  - b. nitric acid.
  - c. acetanilide.
  - d. nitronium ion.
5. Why do you wash the product several times (Procedure Steps 8-10) with ice-cold water?
  - a. to rinse away all unreacted acetanilide.
  - b. to rinse away excess acid.
  - c. a and b are correct.

6. How is the product characterized in this experiment?
  - a. melting point.
  - b. melting point and infrared spectroscopy.
  - c. yield, melting point and infrared spectroscopy.
  - d. none of the above.
  
7. What major differences in absorption bands would you expect to see in the infrared spectra of acetanilide and *p*-nitroacetanilide?
  - a. amide carbonyl adsorption at  $1680\text{ cm}^{-1}$  for both and only a nitro  $\sim 1500\text{ cm}^{-1}$  adsorption at for the starting reagent.
  - b. the absorption due to the introduced nitro group in the product ( $1600\text{-}1500$  and  $1400\text{-}1300\text{ cm}^{-1}$ ).
  - c. the absorption due to the introduced amide group in the product ( $1600\text{-}1500$  and  $1400\text{-}1300\text{ cm}^{-1}$ ).

### Experiment 9 Lab Safety

1. Which reagent(s) used in this experiment must be specially handled, and why?
  - a. nitric acid, it is highly corrosive.
  - b. glacial acetic acid, it is highly corrosive.
  - c. sulfuric acid, it is highly corrosive.
  - d. all of the above.

## Pre-Lab Answers

Exp.1 Prelab Question Answers:

Q1-b; Q2-c; Q3-d; Q4-b; Q5-c; Q6-d; Q7-a; Q8-a; Q9-c; Q10-d

Exp.2 Prelab Question Answers:

Q1-d; Q2-b; Q3-a; Q4-d; Q5-c; Q6-c; Q7-b; Q8-a; Q9-d; Q10-a

Exp.3 Prelab Question Answers:

Q1-b; Q2-c; Q3-c; Q4-d; Q5-c; Q6-b; Q7-e; Q8-c

Exp.4 Prelab Question Answers:

Q1-a; Q2-c; Q3-a; Q4-a; Q5-a; Q6-a

Exp.5 Prelab Question Answers:

Q1-c; Q2-b; Q3-a; Q4-b; Q5-b; Q6-c; Q7-c; Q8-d

Exp.6 Prelab Question Answers:

Q1-d; Q2-c; Q3-d; Q4-d; Q5-b; Q6-a; Q7-b

Exp.7 Prelab Question Answers:

Q1-e; Q2-a; Q3-b; Q4-b; Q5-a; Q6-c

Exp.8 Prelab Question Answers:

Q1-a; Q2-b; Q3-b; Q4-a; Q5-b; Q6-e; Q7-a; Q8-b; Q9-d

Exp.9 Prelab Question Answers:

Q1-c; Q2-b; Q3-d; Q4-c; Q5-b; Q6-c; Q7-b; Q8-d