Queensland Titration Competition 2023

Student Information – University Venues

Section 1. Teams

Entrants are to participate in teams of three.

Section 2. The Task

Each member of a team will be given 2 accurately weighed samples of a solid acid, potassium hydrogen phthalate, which is a primary standard. You will prepare solutions of each of these and perform a set of titrations to determine the amount of acid in the Unknown sample.

Section 3. Apparatus

Exact sizes of beakers and flasks can vary.

For each student:
1 x 25 or 50 mL burette (see Note 3.1)
1 x 20 or 25 mL pipette (see Note 3.1)
1 x 100 mL volumetric flask (see Note 3.2 and 3.3)
4 x 250 mL conical flasks
2 x 100 mL beakers
2 x 250 mL beakers
conical funnel
pipette filler
Pasteur pipettes
stirring rod
wash bottle and supply of deionised/distilled water
retort stand and clamp
Result Sheet (Appendix 1)

The use of self-filling burettes or pipettes, pH meters, automatic titrators, and magnetic stirrers is not permitted.

In keeping with standard laboratory procedures, all competitors should wear appropriate personal protective equipment.

Note 3.l: If a 25 mL burette is used, it is possible that it may require refilling during a titration if the pipette used is also 25 mL.

Note 3.2: If competitors wish to make up the Unknown solution in a second volumetric flask, they are free to do so, but any error in glassware volumes will cancel out only if the same burette, pipette, and volumetric flask are used for both Standard and Unknown Sample titrations.

Note 3.3: If the school does not have a supply of 100 mL volumetric flasks, your teacher will provide you with an alternative method for practice.

Section 4. Chemicals

Each entrant will receive the following:

• A vial ("Standard Sample") containing 0.3000 - 0.5000 g of potassium hydrogen phthalate (KHP) ($C_6H_4COOHCOO^-K^+$ formula mass 204.22) with a label giving an identifying number (e.g. S 15), the mass of sample (to 0.0001 g), and the number of moles of KHP (to 4 significant figures).

• A vial ("Unknown Sample") containing 0.3000 - 0.5000 g KHP labelled with an identifying number (e.g. U 15) (see Note 3.1).

• 300 mL sodium hydroxide solution, about 0.02 mol L^{-1}(All members of a team should have the same concentration of NaOH)

• phenolphthalein

Note 3.1: The Standard and Unknown do not have to have the same numbers, but it is desirable that they are close, as this means that they were probably weighed at the same time.

Section 5. Procedure

• The task for each student is to use 2 sets of acid-base titrations to firstly standardise the solution of NaOH provided, then to find the concentration and thus the **number of moles** of acid in the Unknown Sample**.**

Section 6. Time limits

Two hours are allowed for all work, including calculations. Prior to the official starting time, you may set up and rinse equipment with deionised water only. You may not utilise sample vials or NaOH solution until told to begin.

Section 7. Judging of Results

The competition will be judged on the **number of moles of potassium hydrogen phthalate** recorded on the Result Sheet. See Section 8 for further details.

Since Standard and Unknown Samples with similar numbers are weighed by the same person on the same balance at the same time, results should depend only on the skill of the entrant.

Section 8. Calculating Individual and Team Scores

Deviation: For the result submitted by each individual, round off to 4 significant figures, and multiply by 10^6. For example, result submitted is 2.1034×10^{-3} mol KHP. This becomes 2103. Similarly, multiply the correct value by 10^6. If this were 2.110×10^{-3} mol KHP, it becomes 2110. Subtract (10^6 x correct value) from (10^6 x submitted value) to give the deviation. In this example, the deviation is -7.

The Variance: is the square of the deviation

Team Score: find the sum of **the variance** for each of the 3 team members.

For example, if a team submits the following results:

Name	**Submitted**	**Correct**	**Deviation**	**Variance (deviation)^2**
Bill Smith	2.103×10^{-3}	2.110×10^{-3}	-7	49
Mary Jones	1.907×10^{-3}	1.886×10^{-3}	-21	441
James Wong	1.688×10^{-3}	1.688×10^{-3}	0	0

Team Score 490

Teams will be ranked on this score, with approximately the top 10% of teams being invited to take part in the Finals.

Section 9. Awards

All students will receive a certificate recognising their participation and showing their individual level of achievement.

Certificate levels will be awarded based on the percentage error, according to the following table:

Certificate level	Percentage error
High Distinction	0 – 0.40
Distinction	0.41 – 0.80
Credit	0.81 – 1.20
Competent	1.21 – 1.50
Participation	>1.50

Teams with a Team Score of less than 1000 will be recognised as Excellent Teams. A team Score of 1000 – 1500 will be recognised as a Highly Commended Team.

Members of Excellent and Highly Commended Teams will receive a Certificate stating their team's achievement.

Each school sponsoring one or more Excellent Teams will receive an engraved plaque commemorating the achievement.

Each member of up to 10% of teams participating at any competition venue (providing the team has achieved an Excellent Team standard) may receive an individual plaque commemorating their achievement.

Appendix 1: Result Sheet

ROYAL AUSTRALIAN CHEMICAL INSTITUTE

Queensland Regional Competition 2023

Name _____

Print full name clearly

School _____

Teacher's Name _____

You have been allocated Standard Sample **S** _____

Moles of potassium hydrogen phthalate in Standard Sample (on label) : _____moles

Volume of pipette used _____mL

Volumes of NaOH for individual titrations of Standard Solution (cross out any you have ignored for subsequent calculations)

_____mL _____mL _____mL _____mL

Average volume_____mL Calculated NaOH concentration _____mol L^{-1}

You have been allocated Unknown Sample **U** _____

Volumes of NaOH for individual titrations of Unknown Solution (cross out any you have ignored for subsequent calculations)

_____mL _____mL _____mL _____mL

Average volume _____mL

Calculated moles of potassium hydrogen phthalate in Unknown Sample (4 sig figs e.g. 2.034 x 10^{-3} mol)

_____**moles**

Signature _____

You may use the back of this sheet for rough work.

Appendix 2: Procedure

1. Preliminary preparations

If you cannot clearly read the number of moles of KHP on the Standard label, or if you are uncertain about the identifying numbers, check with a laboratory supervisor who should have a list showing the number of moles in each Standard Sample.

Write your name and other details on the Result Sheet provided.

Rinse all glassware with small volumes of deionised water before the competition time starts.

2. Preparation of Standard Sample

Open the Standard vial (see Note A below) and tip the contents into a beaker. Rinse remaining sample out of the tube into the beaker with deionised water, making sure none of the sample is retained in the lip of the vial, or on the inside of the cap. This requires multiple small rinses.

Add sufficient water to dissolve the sample (20 mL is sufficient) and stir using the glass rod, to dissolve the solid completely.

When the sample is completely dissolved, transfer to a 100 mL volumetric flask, rinsing to ensure that all the dissolved acid is transferred to the flask.

Carefully make up to the 100 mL mark. Stopper the flask, hold your index finger over the stopper, and invert several times to mix thoroughly.

3. Standardising the NaOH solution

Rinse a clean dry beaker with 3 small volumes of your acid solution before transferring the rest of the solution to it.

Rinse your pipette with 3 small volumes of the Standard solution you have prepared.

Pipette out aliquots (accurately measured volumes of the solution – i.e. your pipette volume) into 3 or 4 conical flasks.

Add 3 drops of phenolphthalein indicator to each flask.

Rinse the burette (3 times) with small volumes of the NaOH solution, then fill the burette (see Note B below).

Run the solution from the burette into the flask , swirling to mix until you reach a phenolphthalein endpoint. This should be a pale pink that lasts for 10 seconds before fading. It is important to titrate to a consistent indicator colour.

Repeat the titration as often as time and solution volumes allow.

Calculate the concentration of the sodium hydroxide solution. As all students in a team have the same solution of NaOH, you should compare your NaOH values with those of your team mates. They should be very similar, but not necessarily identical (as your volumetric glassware may not be exactly the same).

4. Rinsing

Rinse your flasks, pipette and volumetric flask with deionised water as in the preliminary instructions. (There is no need to rinse your burette, refill it when you are ready to do the next set of titrations).

5. Titration of the Unknown Solution

Repeat the procedure you used to make the Standard solution, using your Unknown sample.

Repeat the procedure you followed in point 3 above to find the concentration of the Unknown solution.

6. Final calculations

Calculate the number of moles of KHP in the Unknown Sample and enter on the Result Sheet.

Carefully check your calculations. Your answer should be in the range 1.5 – 2.5 x 10^{-3} mol.

Team members should check one another's calculations. Remember that a calculation or transcription error removes any chance of a good result.

Note A: Sometimes it may happen that some of the material adheres to the lid of the vial, and may spill as it is opened. The KHP does tend to "cake" if the vial stands in a particular position for some time. Tapping the lid sharply before the bottle is opened may sometimes assist, but it should always be opened over a beaker as spillages will then usually be contained. Remember always to rinse the lid as well as the inside of the vial.

Note B: When such a dilute sodium hydroxide solution is exposed to the atmosphere, absorption of carbon dioxide can significantly affect the concentration in a short time. A common "symptom" of this problem is that increasing volumes of the sodium hydroxide solution will be required to titrate the same volumes of acid solution. It is important that the second set of titrations is done as soon after the first set as is practicable, as the concentration of the NaOH will decrease slowly over time.

Calculation Guide:

This guide is based on a 20 mL pipette. Adjust for 25 mL if necessary.

Step 1: Concentration of your standard sample

$$\text{concentration of standard} = \frac{\text{no. of moles}}{\text{volume}}$$

where volume = 0.1000L

Step 2: Concentration of NaOH

$$\text{concentration of NaOH} = \frac{\text{concentration of standard x volume of standard}}{\text{volume of NaOH}}$$

(Answer should be approximately 2.0 x 10^{-2} mol.L^{-1})

Step 3: Concentration of Unknown

$$\text{concentration of unknown} = \frac{\text{concentration of NaOH x volume of NaOH}}{\text{volume of acid}}$$

where volume of acid = 20.00 mL

(Answer should be in the range (1.5 – 2.5) x 10^{-2} mol.L^{-1})

Step 4: No. of moles of Unknown

no. of moles of unknown = concentration of unknown x volume of solution

where volume of acid = 0.1000L

(Answer should be between 1.5 x 10^{-3} and 2.5 x 10^{-3} moles.)

Check formula:

$$\text{no. of moles of unknown} = \text{no. of moles in standard sample} \times \frac{\text{volume of NaOH used to neutralise unknown}}{\text{volume of base used to neutralise standard}}$$

Appendix 3 Summary of Procedure

Standardisation

1. Rinse **all** glassware 3 times with DI water.
2. Dissolve Standard Sample in a small beaker.
3. Transfer Standard Sample to volumetric flask (lots of rinsing), and add DI water to fill to the line.
4. Rinse a clean beaker with small amounts of the Standard solution.
5. Transfer Standard solution to rinsed beaker.
6. Rinse pipette with Standard solution.
7. Pipette out aliquots (accurately measured volume of the solution) into flasks. Add indicator to each.
8. Rinse and fill burette with NaOH solution.
9. Conduct titration and repeat for all aliquots, refilling burette as required.

Analysis of Unknown

1. Rinse all glassware except burette with DI water.
2. Dissolve Unknown Sample in small beaker.
3. Transfer Unknown Sample to volumetric flask (lots of rinsing).
4. Rinse a beaker with small amounts of the Unknown solution.
5. Transfer Unknown solution to rinsed beaker.
6. Rinse pipette with Unknown solution.
7. Pipette out aliquots into flasks. Add indicator to each.
8. Refill burette with NaOH solution.
9. Conduct titration and repeat for all aliquots, refilling burette as required.