Australian National Titration Competition 2022

Finals Details – University Venues

Section 1. Teams

Entrants are to participate in the Finals in teams of three. These should, if possible, be the same teams as have performed well in the Regional Competition. Substitution of others from the same school is permitted (e.g. in cases of sickness) but is not encouraged.

Section 2. Apparatus

The following apparatus should be available, either brought by the team or supplied by the university, as determined in consultation with the Regional Organiser. Exact sizes of beakers and flasks can vary.

For each student:

• 1 x 25 or 50 mL burette (see Note 2.1)

• 1 x 20 or 25 mL pipette (see Note 2.1)

• 1 x 100 mL volumetric flask (see Note 2.2)

• 4 x 250 mL conical flasks

• 2 x 250 mL beakers

• 2 x 100 mL beakers

• conical funnel

• Pasteur pipettes

• pipette filler**

• glass rod

• wash bottle and supply of deionised/distilled water

• retort stand and clamp, filter stand

• Result Sheet (Appendix 1)

**** Teachers must instruct their students about safe insertion of pipettes into fillers.**

For each team:

• supply of filter papers (see Note 2.3 below)

• marking pens or labels

The following will not usually be supplied by the Organiser, but may be brought by the entrants: calculators, magnifying glasses, written material, simple pipette fillers.

Use of self-filling burettes or pipettes, pH meters, automatic titrators, and magnetic stirrers is not permitted. Filtration is by gravity (not suction).

In keeping with standard laboratory procedures, all competitors must wear appropriate personal protective equipment. Teachers should take the relevant PPE for their students to the university venue.

Note 2.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 2.2: 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 2.3: The barium sulfate in the Unknown Sample should be retained by Whatman #1 papers, provided that the filtration is carried out correctly. Local Organisers may, at their discretion, supply finer (slower) paper, or teams may supply their own.

Section 3. Chemicals

Each entrant will receive the following samples:

A vial ("Standard Sample") containing 0.3000 - 0.5000 g of potassium hydrogen phthalate (KHP - formula mass 204.22) with a label giving an identifying number (e.g. S152), 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 mixed with barium sulfate (0.05 - 0.15 g) labelled with an identifying number (e.g. U154) (see Note 3.1).

A screw-cap or stoppered bottle containing approx. 300 mL NaOH solution, about 0.02 mol L^{-1} should be available to each team member, **with a different concentration for each team member.**

A bottle of **fresh** phenolphthalein indicator solution.

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

Section 4. Procedure

After standardising the sodium hydroxide solution against a solution of their Standard Sample, each member of the team will analyse one Unknown Sample. Each competitor is free to carry out any procedure with the apparatus provided in the time allowed. While Appendix 2 details the "Orthodox Procedure", adherence to it is not compulsory. It has been shown over the years that the competition has run that, carefully carried out, it can give excellent results. Some teams have also obtained excellent results by using variations on this procedure.

Section 5. Time limits

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

Section 6. Replacement samples

Replacement samples are provided at the discretion of the Organiser, who will have a limited number. Replacements are given only if there is an accident or mishap - not, in general, if an entrant simply runs out of solution, or goes "over the mark" of the volumetric flask. If a replacement sample is used, make sure you provide the new sample number on your Result Sheet.

Section 7. Judging of Results

The competition will be judged on the accuracy of the number of moles of potassium hydrogen phthalate written on the Result Sheet.

National rankings for Excellent and Highly Commended teams only will be available after all results from State Finals have been communicated to the National Coordinator in late September.

Since Standard and Unknown Samples with the same numbers are weighed by the same person on the same semi-micro balance (to 5 dec. places) consecutively, results should depend only on the skill of the entrant.

Section 8. Calculating Individual and Team Scores

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 x 10^{-3} mol KHP. This becomes 2103. Similarly, multiply the correct value by 10^6. If this were 2.110 x 10^{-3} mol KHP, it becomes 2110. Subtract (10^6 x correct value) from (10^6 x submitted value) to give (10^6 x deviation) or $10^6\Delta$. In this example, $10^6\Delta$ is -7. If the absolute value of this number is 20 or less, the entrant receives a *gold* award; otherwise a *silver* award.

For a team, **square the value of $10^6\Delta$** for each team member, and add the three values of $10^{12}\Delta^2$ for the team members to give the team's "score". If the score is 1000 or less, the team is an *Excellent Team*. If the score is between 1001 and 1500 (incl), it is a *Highly Commended* Team. Only details of Excellent and Highly Commended Teams need to be provided to the National Coordinator.

For example, if a team submits the following results:

Name	**Submitted**	**Correct**	**$10^6\Delta$**	**$10^{12}\Delta^2$**	**Individual Award**
Bill Smith	2.103 x 10^{-3}	2.110 x 10^{-3}	-7	49	Gold
Mary Jones	1.907 x 10^{-3}	1.886 x 10^{-3}	-21	441	Silver
James Wong	1.688 x 10^{-3}	1.688 x 10^{-3}	0	0	Gold

Team Score 490 This would be an Excellent Team.

Section 9. Awards

Each student participant will receive either a gold or a silver badge as detailed in Section 8 above.

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 teams placed First, Second and Third will receive an individual plaque commemorating their achievement.

The winning school will hold the Trevor Appleton Memorial Trophy for a period of 1 year. The name of the winning school will be engraved upon this each year.

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ROYAL AUSTRALIAN CHEMICAL INSTITUTE

Royal Australian Chemical Institute

Australian National Titration Competition

National Finals Result Sheet

Name _____ Team No_____

Print your name clearly

School _____

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**

Correct Answer	= . . . . .
Δ	= . . . . .
Team score $\Sigma \Delta^2$	= . . . . .

Appendix 2: Orthodox Procedure

1. Rinse all glassware with small volumes of deionised water.

2. Carefully examine the samples provided. If there is any sign of damage to the lids of the vials, or of spillage, request a replacement. 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 the organiser 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.

3. Open the Standard vial (see Note A), and tip the contents into a beaker. Rinse remaining sample out of the tube into the beaker with deionised water, making sure none is retained in the lip of the vial, or on the inside of the cap. Rinse the vial a number of times, transferring the rinsings to the beaker. Add sufficient water to dissolve the sample (but much less than 100 mL total) and stir using the glass rod, to dissolve the solid completely. When the sample has all dissolved, transfer to a 100 mL volumetric flask, rinsing multiple times to ensure that all of the dissolved acid is transferred to the flask. Carefully make up to the 100 mL mark. Stopper the flask and invert several times to mix thoroughly.

4. Rinse a clean beaker with small amounts of the solution before transferring the solution to it. Rinse the pipette with 3 small volumes of the solution. Pipette out 20 or 25 mL into a conical flask. Add 3 drops of phenolphthalein indicator. Repeat this to obtain 3 or 4 flasks of acid plus indicator ready to titrate.

5. Rinse the burette (3 times) with small volumes of the sodium hydroxide solution, then fill the burette (see Note B). Run the solution from the burette into the flask to a phenolphthalein end-point. This should be a pale pink that lasts for 10 seconds before fading. For dilute solutions such as these, it is important to titrate to a consistent indicator colour. Repeat the titration as often as time and solution volumes allow.

6. Calculate the concentration of the sodium hydroxide solution.

7. Dissolve the KHP in the Unknown Sample in deionised water in a small beaker, and filter the solution to remove $BaS0_4$, remembering to rinse the beaker thoroughly. The efficiency with which the paper is rinsed is important, but remember that the total volume of filtrate plus washings must not exceed 100 mL. Transfer the solution to a volumetric flask (rinsing to ensure that all of the dissolved acid is transferred to the flask) and make up as for the Standard.

8. Repeat Steps 4 and 5 above using your Unknown solution.

9. 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 $(1.5 – 2.5) \times 10^{-3}$ 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 if 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 or flask with a funnel in the neck, as spillages will then usually be contained. Remember always to rinse the lid as well as the inside of the vial. The Organiser usually has a few spare samples, and a replacement sample may be appropriate if there is spillage at this stage.

Note B: If 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.