



Australian National Titration Competition

The Competition has been conducted throughout Australia since 1984, with approximately 150 teams taking part in the National Finals each year. In 2014 the Dr Trevor G Appleton Memorial Award was commissioned. The school that wins the Competition will hold this Award for a year.

Teams that have achieved highly in their Regional Titration Competition are invited to take part in the National Finals of the Competition. Regional Coordinators will inform teams' supervising teachers of the local details. In most metropolitan regions, the Regional Coordinator will liaise with schools to run the Competition at a central venue. In other regions, teachers liaise directly with the National Coordinator who sends samples to their school. These teams complete the analysis on their school campus.

The three hour task involves two weighed samples of a solid acid (potassium hydrogen phthalate). The Standard Sample is used to standardise a dilute solution of sodium hydroxide. The difficulty of the analysis is increased by the addition of a small amount of insoluble barium sulfate to the Unknown Sample. Teams are judged on the accuracy with which each member determines the amount of acid present in the Unknown Sample. Detailed information is provided in the Competition Details.

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Finals Details & Rules

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, 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)
- 2 x 250 mL conical flasks
- 1 x 250 mL beaker
- 1 x 100 mL beaker
- Conical funnel
- Pasteur pipettes
- Glass rod
- Wash bottle and supply of deionised/distilled water
- Retort stand and clamp, filter stand
- Result Sheet (Appendix 1)

For each team:

- Supply of filter papers (see Note 2.3 below)
- Bottle of phenolphthalein indicator solution
- 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. **If pipette fillers are to be used, teachers must instruct their students about safe insertion of pipettes into fillers, before they come to the competition venue.**

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, appropriate personal protective equipment should be worn by all competitors.

Note 2.1: 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 Finalists 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 bring some with them.

Section 3 Chemicals

Each entrant will receive the following samples:

- A sample tube ("Standard Sample") containing 0.3000 - 0.5000 g of potassium hydrogen phthalate (KHP - formula mass 204.22) with a label giving the mass of sample (to 0.0001 g), the number of moles of KHP, and an identifying number (e.g. S152).
- A sample tube ("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 sodium hydroxide solution, about 0.02 M should be available to each team member, with a different concentration for each team member.

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 weighed at the same time.

Section 4 Procedure

Each member of the team will analyse one Unknown Sample, after standardising the sodium hydroxide solution against the Standard Sample. Each Finalist is free to carry out any procedure with the apparatus provided in the time allowed. 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 distilled water only. They may not utilise sample vials or base 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 number of moles of potassium hydrogen phthalate written on the Result Sheet. This value will be subtracted from the correct value, and the difference (Δ) then squared. These squares (Δ^2) will be added for the three members of each team. The winning team is the one for which the sum of Δ^2 is the smallest.

At every venue where teams from more than one school participate, the Organiser will receive a sealed envelope listing the correct results for the Unknown Samples sent to that venue. The envelope may be opened at the end of the Regional Finals Competition, so that each Finalist will know how good the individual performance is, and whether the team meets the criteria for "Excellent" or "Highly Commended" Team (see Section 8 below). National rankings for "Excellent" and "Highly Commended" teams only will be available after all results from Regional Finals have been communicated to the National Coordinator in late October.

Where teams compete on their own campus under teacher supervision, student results are to be sent (by email) directly to the National Coordinator who will then judge them and provide feedback to the teacher. 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

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 ($10^6 \times$ correct value) from ($10^6 \times$ submitted value) to give ($10^6 \times$ 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' plaque; otherwise a 'silver' plaque.

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, 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 Plaque
Bill Smith	2.103×10^{-3}	2.110×10^{-3}	-7	49	Gold
Mary Jones	1.907×10^{-3}	1.886×10^{-3}	-21	441	Silver
James Wong	1.688×10^{-3}	1.688×10^{-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' plaque 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 an Excellent Team 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 one (1) year. The name of the winning school will be engraved upon this each year.

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Appendix 1: National Finals Result Sheet

Name: _____
Print full name clearly

School: _____

School Address: _____ Postcode: _____

Teacher's Name: _____

Centre for Finals Competition: _____

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

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

Pipette Volume _____ mL

Volumes of NaOH for individual titrations of Standard Solution (place X against any you have ignored for subsequent calculations)

_____ mL _____ mL _____ mL _____ mL

Average volume _____ mL Calculated NaOH Molarity _____ M

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

Volumes of NaOH for individual titrations of Unknown Solution (place X against any you have ignored for subsequent calculations)

_____ mL _____ mL _____ mL _____ mL

Average volume _____ mL

Calculated Moles of potassium hydrogen phthalate in Unknown Sample (4 significant figures)

_____ Moles

Signature: _____

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

Appendix 2: Orthodox Procedure

Carefully examine the samples provided. If there is any sign of damage to the lids of the bottles, 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.

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 distilled water, making sure none is retained in the lip of the vial, or on the inside of the cap. Add sufficient water to dissolve the sample (but less than 100 mL total) and stir using the glass rod, to dissolve the solid completely (alternatively, the sample could be transferred with the aid of a funnel into a conical flask, which is stoppered and shaken vigorously). When the sample has all dissolved, transfer to a 100 mL volumetric flask, and carefully make up to the mark. Stopper and mix well. Pipette out 20 or 25 mL into a conical flask. Add phenolphthalein indicator.

Rinse the burette with some 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. For dilute solutions, 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.

Dissolve the KHP in the Unknown Sample in distilled water, and filter the solution to remove BaSO_4 (see Note 2). 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 and make up as for the Standard. Pipette out aliquots, and titrate as before.

Calculate the number of moles of KHP in the Unknown Sample, and enter on the Result Sheet. Carefully check your calculations. Team members may 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.