Australian National Chemical Analysis Competition - Finals Rules

1. Entrants should 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 not encouraged.

2. Each member of a team should have at least the following apparatus, either brought by the team or supplied, as determined in consultation with the Regional Organiser. Exact sizes of beakers and flasks can vary.

1 x 25 or 50 mL burette (see note 1)
1 x 20 or 25 mL pipette (see note 1)
1 x 100 mL volumetric flask (see note 2)
2 x 250 mL conical flasks
1 x 250 mL beaker
1 x 100 mL beaker
conical funnel
medicine dropper bulb and several droppers
wash bottle and supply of deionised/distilled water
stand and clamp, filter stand
Results Sheet

For the team:

supply of filter papers (see note 3)
marking pens or labels

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.
One bottle of phenolphthalein indicator solution will be supplied to each team.

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).

3. 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 4).

4. 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. The following is 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.

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 5), 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 make up carefully to the mark. Shake well to ensure complete mixing. 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 6). 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 desired, 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 $BaS0_4$ (see note 3). 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 (see note 2 - if you wish to keep the solution that was in the flask, store in a labelled flask or beaker). 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.

5. Three hours are allowed for all work, including calculations.

6. Replacement samples are provided at the discretion of the Organiser, who will usually have only a limited number. Replacements are usually 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 that it is the new sample number which appears on your Result Sheet.

7. 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) 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.

8. 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 below). National rankings will be available after all results from Regional Finals have been communicated to me in late October.

9. 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 (and luck) of the entrant.

Note 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. 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 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.

Note 4. 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.

Note 5. 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 6. 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.

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 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' medal; otherwise a silver medal.

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 1000 and 1500, it is a "Highly Commended" Team. Only details of Excellent and Highly Commended Teams need to be transmitted to me.

For example, if a team submits the following results:

Name	Submitted	Correct	$10^6\Delta$	$10^{12}\Delta^2$	Medal
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".			

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