

Experimental Techniques in Chemistry

A Teacher's Guide

Hong Kong Examinations Authority, 1987

Introduction

This guide has been written to complement the four video programmes on experimental techniques produced by a working party of the Hong Kong Examinations Authority. The titles of the video programmes are:

1. Carrying Out the Flame Test
2. Use of Quickfit Apparatus
3. Acid-Base Titration
4. Active Ingredients in Aspirin Tablets

The guide indicates the teaching points illustrated in the programmes and considers the areas where assessment would be appropriate. Both the teacher's guide and the programmes have been produced by a working party, in response to concerns expressed by teachers on the teaching and assessment required in A-level Chemistry practical work.

It is expected that teachers will wish to show each programme either at one time or in parts, and they have been produced with this in mind. The programmes illustrate techniques that are expected of students and hence lay stress on the manipulation of apparatus, carrying out experimental procedures, taking readings, making observations, safety aspects and general bench performance.

Teaching points

Each guide lists the teaching points related to the experimental techniques. The techniques shown in the programmes may not concur with those currently used in your teaching and the working party is aware of other alternatives in experimental techniques which are equally acceptable. These notes, whilst not claiming to be exhaustive, are written to cover major teaching criteria and it is hoped that all teachers will cover these in their practical lessons. It must be remembered that assessment of manipulative and observational skills is not appropriate before students have received the relevant teaching.

The guide also points out relevant planning and interpretative skills. The programmes are unable to show these, yet they also form an important aspect of teaching.

Assessment Points

The main purpose of the guide is to assist teachers with the assessment of practical work. In the guide, assessment points related to assessment of Ability Area A have been given separately, under the criteria:

1. Operation of a single piece of apparatus
2. Carrying out experimental procedures
3. Reading and measurements of different instruments/ apparatus
4. Observational skills
5. General bench performance

These criteria have been chosen, based on the reactions of teachers to the assessment plan put forward by one member of the working party. In the plan, manipulative skill assessment can be divided into five aspects (1 to 5 above), although not all five would be applicable to a given experimental situation. Further details of the plan can be obtained from the working party.

Assessment points for Ability Area B have also been given, although interpretative skill assessment is not possible in some cases. Attention is drawn to the assessment of planning skills as this is an area where problem-solving skills in practical work can be assessed cognitively by writing out a plan, or, in terms of manipulative skills, by carrying out the experiment planned.

It is the wish of the working party that teachers should take note of the assessment guidelines proposed. There is the implicit suggestion that if all teachers follow such guidelines, then this would serve as a powerful indicator of standards across Hong Kong schools and hence help to increase the reliability of assessment under the Teacher Assessment Scheme.

Programme 1 : Carrying Out The Flame Test

Main Objective: To identify criteria for the assessment of techniques in carrying out the Flame Test.

Duration: 4 1/2 minutes

Criteria for Assessment:

A. Manipulative skills, observation and general bench performance

1. Operation of a single piece of apparatus

Assessment Point	Teaching Point
<i>Students should be able to</i> (a) use the Bunsen Burner	use the Bunsen Burner by (i) regulating the gas flow (ii) adjusting the air-hole (iii) using a non-luminous flame in conducting the Flame Test
(b) use a piece of cobalt blue glass	view a flame through the cobalt blue glass while introducing the platinum/nichrome wire into the flame

2. Carrying out experimental procedures

Assessment Point	Teaching Point
<i>Students should be able to</i> (a) clean a platinum / nichrome wire properly	clean a platinum/nichrome wire by

	<ul style="list-style-type: none"> (i) moistening the wire with concentrated hydrochloric acid placed in a watch glass/test-tube (When using concentrated hydrochloric acid, care should be taken to keep one's face away from the acid fumes, and to limit the time the stopper is out of the bottle. Any spillage should be cleaned immediately with a moist cloth.) (ii) introducing the moistened wire into the tip of the blue cone of a roaring Bunsen flame (iii) repeating (i) and (ii) if necessary until there is no flame coloration
(b) conduct the Flame Test	<p>conduct the Flame Test by</p> <ul style="list-style-type: none"> (i) putting a small amount of the powdered sample onto a watch glass or other suitable container (ii) moistening the cleaned wire with a fresh supply of concentrated hydrochloric acid (iii) dipping the tip of the moistened wire into the powdered sample, so that some of it adheres to the wire (iv) introducing the tip of the wire to the base of a quiet, non-luminous Bunsen flame, gradually moving the wire to a hotter part of the flame if no colour is observed (v) repeating (ii) to (iv) for further confirmation, if necessary

3. Observational skills

Assessment Point	Teaching Point
<p><i>Students should be able to</i></p> <p>(a) observe and record the characteristic flame colours</p>	<p>observe and record the characteristic flame colours, eliminating those that come from impurities</p> <p>observe that some flame colours may</p> <p>(i) show up later than others</p> <p>(ii) be more persistent than others</p>

4. General bench performance

Assessment Point	Teaching Point
<p><i>Students should be able to</i></p> <p>(a) take safety precaution when carrying out Flame Tests</p> <p>(b) utilise available bench space sensibly</p> <p>(c) keep the working area tidy</p> <p>(d) use the time available efficiently</p>	<p>take safety precaution by</p> <p>(i) wearing safety goggles</p> <p>(ii) paying attention to the handling of concentrated hydrochloric acid, so as to minimize the amount of acid fumes and to mop up any spillage immediately</p>

<p>(e) take safety precautions in using the Bunsen Burner</p>	<p>take safety precautions in using the Bunsen Burner by</p> <p>(i) not leaving a non-luminous flame unattended</p> <p>(ii) adjusting to a luminous flame when the burner is not in use</p> <p>(iii) turning off the gas supply should “striking back” occur, and relighting only when the burner is cool</p>
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B. Planning the experiment and interpretation of data

Assessment Point	Teaching Point
<p><i>Students should be able to</i></p> <p>(a) identify the common cations by their flame colour, volatility and persistency, etc.</p> <p>(b) decide whether cobalt blue glass should be used in the identification of potassium ions in the presence of sodium ions</p>	

Programme 2: Use of Quickfit Apparatus

Main Objective: To identify criteria for assessing basic techniques in the assembly and use of quickfit apparatus.

Duration: 8 1/2 minutes

Criteria for Assessment

A. Manipulative skills, observation and general bench performance

1. Operation of a single piece of apparatus

Assessment Point	Teaching Point
<i>Students should be able to</i>	lubricate the glass joint evenly, using only a small amount of lubricant clamp the apparatus (i) at a suitable position (ii) with the movable parts uppermost (iii) firmly but not too tight
(a) place the thermometer bulb in the correct position	adjust the position of the thermometer by unscrewing the adapter, sliding the thermometer through the ring and then retightening it set up a water condenser by (i) checking that the rubber tubing is of sufficient length (ii) fitting the rubber tubing by lubricating with a little water
(b) run water into the condenser from the lower end	(iii) running water into the condenser from the lower end, and

(c) control the rate of flow of water in the condenser	(iv) controlling the rate of flow of water so that there are no air bubbles, the tubing is not dislodged, and the rate is geared to the boiling point of the distillate
(d) check that the side arm of a T-shaped adapter is pointing upwards	check that when a T-shaped adapter is used for connecting a receiver flask, the side arm of the adapter should be pointing upwards

2. Carrying out experimental procedures

Assessment Point	Teaching Point
<i>Students should be able to</i>	
(a) assemble the apparatus in a systematic manner, without the need to frequently dismantle and re-adjust the position of the apparatus	assemble the apparatus by (i) considering the position of clamping, distance from the sink/ burner, etc. (ii) starting from the base and building up from there
(b) introduce chemicals without having any chemicals left at the joints	transfer chemicals into the reaction flask without having any chemicals sticking to the joints
(c) assemble the apparatus correctly	check, after assembling the apparatus, that (i) the system is not a closed one (ii) the joints are well fitted (iii) the system is not tilted
(d) add anti-bumping granules correctly	use anti-bumping granules by (i) adding only a few (ii) adding them only when the liquid is cold
(e) avoid over-heating	adjusting the rate of heating by (i) controlling the size of the flame/ temperature of the heater (ii) removing the burner at suitable time(s)

(f) collect the distillate properly	collect the distillate (i) at a suitable rate (ii) within a suitable temperature range (iii) in a receiver that is adequately supported and correctly positioned dismantle the apparatus as soon as it is cool enough to be handled clean the apparatus properly
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3. Reading and measurement of different instruments/apparatus

Assessment Point	Teaching Point
<i>Students should be able to</i> (a) read and record the temperature/ temperature range of the distillate/ various distillate fractions (to the nearest °C)	read and record the temperature/ temperature range of the distillate/ various distillate fractions (to the nearest °C)

4. Observational skills

Assessment Point	Teaching Point
<i>Students should be able to</i> (a) detect any irregularities as they arise	detect any irregularities as they arise, e.g. (i) leakage at the joints (fumes, bubbling at the joints) (ii) lack of reaction in the flask

5. General bench performance

Assessment Point	Teaching Point
<i>Students should be able to</i> (a) avoid having unnecessary apparatus lying on the bench	avoid having unnecessary apparatus lying on the bench, e.g. the boxes for holding the quickfit sets, unused rubber tubing, etc.

B. Planning the experiment and interpretation of data

Assessment Point	Teaching Point
<i>Students should be able to</i> (a) plan how to carry out an experiment efficiently	plan how to carry out an experiment by (i) deciding on the appropriate quantities of chemicals to be used (ii) choosing suitable apparatus for carrying out a particular process (iii) designing a workable procedure consider the various facilities required, e.g. location of the sink, heating devices, etc., to avoid unnecessary dismantling and re-adjustment

Programme 3: Acid-Base Titration

Main Objective: To identify criteria for assessing basic techniques in carrying out acid-base titrations.

Duration: 19 minutes

Criteria for Assessment:

A. Manipulative skills, observation and general bench performance

1. Operation of a single piece of apparatus

(a) The pipette

Assessment Point	Teaching Point
<i>Students should be able to</i>	
(a) rinse a pipette	rinse a pipette with a small amount of the solution to be titrated - draining it only through the tip
(b) keep the tip of the pipette well below the surface of the solution during pipetting	keep the tip of the pipette well below the surface of the solution during pipetting wipe the outside of the pipette
(c) control the amount of solution in the pipette	control the amount of solution in the pipette by regulating the pressure inside it through minute movement of the forefinger on the top end of the pipette stem ensure that the bulb of the pipette is not held in the hand, especially when filling with solution

(b) The pipette filler

Assessment Point	Teaching Point
<i>Students should be able to</i>	
(a) use a pipette filler properly	use a pipette filler (i) whenever a pipette is to be filled (ii) by following the correct sequence of opening the valves for a filler with valves (iii) and ensure that no liquid enters the bulb
(b) deliver the exact quantity of liquid from a pipette	deliver the bulk of the liquid into a conical flask, the final portion being drained out by touching, for a few seconds, the bottom of the flask in a tilted position, by touching the liquid surface, or by touching the side of the conical flask

(c) The Burette

Assessment Point	Teaching Point
<i>Students should be able to</i>	
(a) use a burette	use a burette by ensuring that (i) after filling the burette, the filter funnel (if used) is removed (ii) there are no air bubbles trapped in the liquid column, and the space from below the stopcock to the tip is filled with solution (iii) the burette is clamped in an upright position, and the burette reading is taken with the meniscus at eye level

2. Carrying out experimental procedures

Assessment Point	Teaching Point
<i>Students should be able to</i>	
(a) prepare a solution in a volumetric flask without spillage and without overshooting the graduation mark	prepare a solution in a volumetric flask by <ul style="list-style-type: none"> (i) dissolving the solute in the minimum amount of solvent in a beaker (ii) washing the volumetric flask with the solvent before filling with the solution (iii) transferring the solution to the volumetric flask without spillage, and without overshooting the graduation mark (iv) making up to the mark by adding the last drops with a dropper, a glass rod, or from the wash bottle (v) ensuring homogeneous mixing of the prepared solution by inverting the volumetric flask several times
(b) prepare the pipette/burette for use	prepare the pipette/burette for use by <ul style="list-style-type: none"> (i) rinsing first with distilled water, and then with the liquid to be used (ii) draining the liquid out of a pipette through the jet (iii) draining the liquid from a burette by inverting it with the tap closed, then opening the tap while draining the liquid from the column
(c) carry out the titration process	carry out the titration process by <ul style="list-style-type: none"> (i) transferring the solution from a volumetric flask to a beaker rinsed with the solution

	<ul style="list-style-type: none"> (ii) pipetting a fixed volume of solution to a clean conical flask (Not rinsed with the solution) (iii) adding a suitable quantity of the indicator to be used (iv) placing the conical flask on a white tile, and then lowering the burette to a position such that the tip is just below the rim of the conical flask (v) adjusting the rate of addition of liquid by controlling the stopcock or the tap, so that the liquid can be added continuously, or one drop at a time, as appropriate (vi) swirling the conical flask during the addition of liquid from the burette to ensure thorough mixing (vii) washing down any liquid adhering to the inner wall of the conical flask with distilled water
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3. Reading and measurement of different instruments/apparatus

Assessment Point	Teaching Point
<i>Students should be able to</i>	
(a) view the meniscus on a graduation mark at eye level	view the meniscus on a graduation mark at eye level
(b) record burette readings accurately	record burette readings to two decimal places, the second decimal place being obtained by estimation

4. Observational skills

Assessment Point	Teaching Point
<i>Students should be able to</i> (a) detect the end-point of a titration	detect the end-point of a titration by noting the correct colour changes in the solution

5. General bench performance

Assessment Point	Teaching Point
<i>Students should be able to</i> (a) transfer liquids without spillage (b) prevent the pipette from rolling on the bench	

B. Planning the experiment and interpretation of data

Assessment Point	Teaching Point
<i>Students should be able to</i> (a) plan an experiment involving titration techniques (b) interpret experimental data	plan an experiment involving titration techniques by choosing the appropriate apparatus, materials, indicator(s), and method of working interpret the experimental data obtained, and make use of the data to perform relevant calculations

(c) obtain and record results to an acceptable degree of accuracy	obtain and record results to an acceptable degree of accuracy by (i) selecting consistent titres for calculating the average titre, e.g. $\pm 0.01 \text{ cm}^3$, or according to a specified level of accuracy (ii) calculating molarity to 4 decimal places (iii) calculating percentage purity to the nearest 1% (iv) determining masses to $\pm 0.001 \text{ g}$
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Programme 4: Active Ingredients In Aspirin Tablets

Main Objective: To identify criteria for assessing basic techniques in crystallisation and melting point determination.

Duration: 12 1/2 minutes

Criteria for Assessment:

1. Operation of a single piece of apparatus

Assessment Point	Teaching Point
<i>Students should be able to</i>	
(a) use a mortar and pestle	use a mortar and pestle to grind crystals into a powder
(b) use filter paper in filtration	fold a fluted filter paper
(c) use a filter funnel	use a filter funnel for filtering hot solutions
(d) use a melting point apparatus	use a melting point apparatus

2. Carrying out experimental procedures

Assessment Point	Teaching Point
<i>Students should be able to</i>	
(a) set up a water bath	set up a water bath by considering <ul style="list-style-type: none"> (i) the size of the container (ii) the quantity of water in the bath (iii) the rate of heating necessary to maintain the bath at a suitable temperature

(b) dissolve a solid in the minimal amount of solvent	dissolve a solid by <ul style="list-style-type: none"> (i) choosing an appropriate solvent (ii) using the minimal amount of solvent (iii) crushing the solid to a powder form in a mortar and pestle if necessary (iv) taking any necessary precautions against fire hazards when flammable solvents are used
(c) filter a hot, saturated solution	filter a hot, saturated solution by <ul style="list-style-type: none"> (i) using a piece of folded filter paper (ii) ensuring that the crystals do not form during the filtration process
(d) carry out the process of suction filtration	carry out the process of suction filtration by <ul style="list-style-type: none"> (i) using a Hirsh/Buchner funnel, moistening the filter paper with solvent to ensure proper suction (ii) using a Buchner flask and a suction pump (iii) ensuring that the filtrate is not sucked into the pump by adjusting the direction of the tip of the funnel (iv) washing the crystals with the minimal amount of cold solvent
(e) prepare a melting point tube	prepare a melting point tube by <ul style="list-style-type: none"> (i) using a capillary tube of suitable length (ii) sealing one end by heating in the flame with a rotating motion

(f) determine the melting point of a purified sample	determine the melting point of a purified sample by (i) drying the crystals thoroughly in a desiccator/oven before introducing into the melting point tube (ii) packing the solid into a melting point tube by gently tapping it, or by dropping it through a glass tube
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