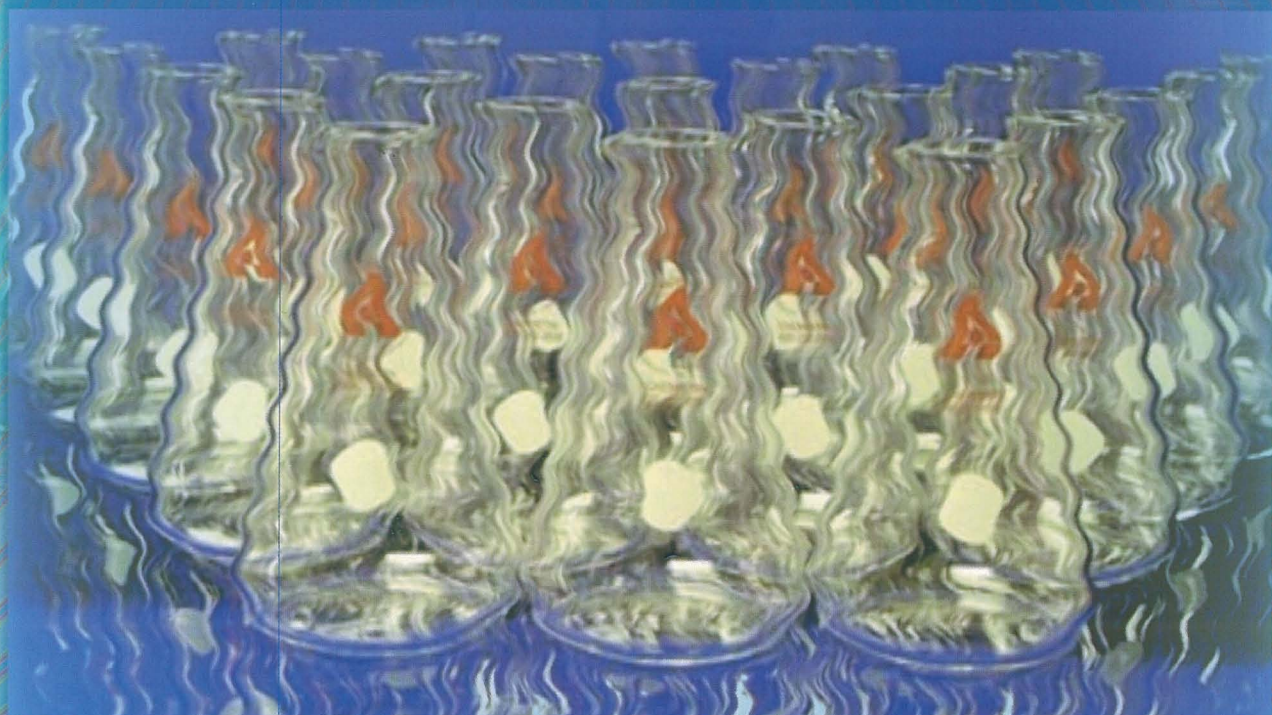


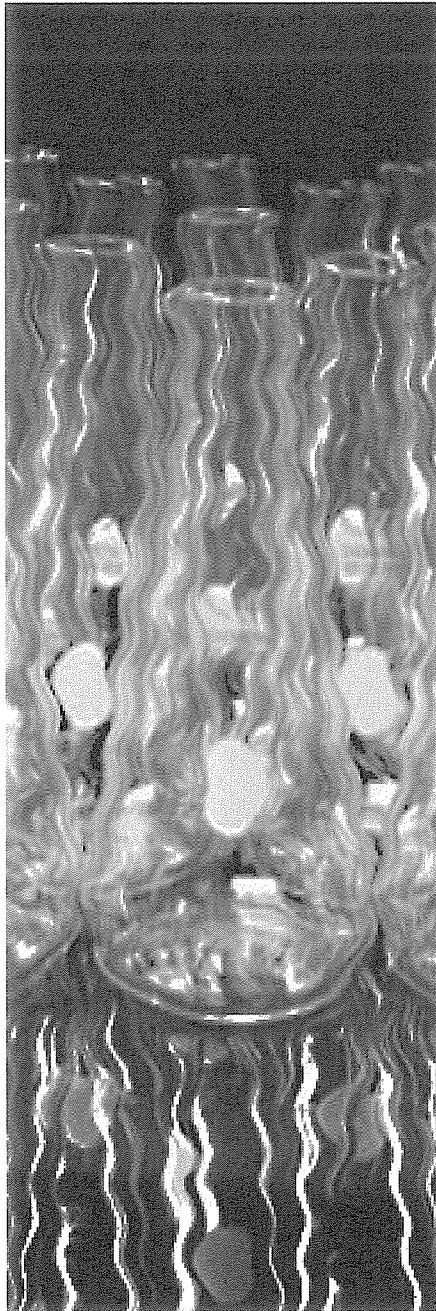


The Association
for Science Education

School and
College
Laboratory
Technicians'
Guide



PREPARING SOLUTIONS



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Laboratory
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Guide



The **Association**
for **Science Education**

Introduction

It has long been an aim of the ASE Laboratory Technicians' Committee to provide some help within the area of training. The results of the nationwide technician survey carried out in 2001 by ASE and The Royal Society* showed that 35% of practising technicians had never been on a training course, with just 31% having attended a chemical handling course. Reasons such as a decreasing number of suitable courses, rising costs and time and workload constraints have been given. This new series of Technicians' Guides aims to go some way to redressing the balance.

Each Technicians' Guide covers one topic and provides background information, details of useful procedures and health and safety advice, to support the excellent service offered by technicians to school and college science.

The contents will be particularly useful for new technicians, for those more experienced technicians guiding them and for those working towards qualifications such as S/NVQ. The design of the Guides, with each topic being made available singly and shrink-wrapped, will allow technicians to build up their own handbook of operating procedures, adding in new titles as they are published.

**Survey of Science Technicians in Schools and Colleges (The Royal Society/ASE, 2001).*

Preparing Solutions

This set of procedures can be used by experienced technicians to remind them of good practice and by new technicians as a means of increasing their experience and expertise. This is one of the most essential jobs a technician carries out.

ASE Laboratory Technicians' Committee

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1 Introduction

It is convenient to keep stock solutions of common reagents. These may be quite large volumes – 2.5 or 5 dm³ or even more, sometimes in aspirators. On receipt of practical requests from teachers, the stock solutions can be diluted to the concentration required for the procedures to be carried out by the students or for teacher demonstration. A risk assessment is required to cover carrying out this dilution.

Request sheets and/or schemes of work from teachers should always specify the concentration and volume required for a practical session. Health and safety references should also be included. Model risk assessments should be available.

Students are learning to use chemicals and will make mistakes. They will have to repeat a procedure. They will pour more than required into beakers and test tubes; they will spill solutions. When providing solutions for practical work, the technician should prepare more than the teacher estimates. (If the instructions suggest 10 cm³ per group and there are 15 groups then prepare at least 300 cm³.) Commonly needed solutions will be prepared in advance and stored.

1.1 Stock solutions and 'bench' solutions

Stock solutions are kept in the stores and are available for further dilution. 'Bench' solutions are so named because traditionally a considerable amount of qualitative analysis was carried out in sixth forms. Students had to have immediate access to a range of solutions in the laboratory (and 30 years ago these were on the bench!). In the specifications used now, they are not required.

However, there may be a need for solutions to be stored in laboratories situated some distance from a prep room. In such cases, the solutions should be kept secure in a locked cupboard and brought out as and when requested by the schemes of work.

1.2 Hazards and risk assessment

Concentrated acids and alkalis are usually corrosive and/or toxic and some give off fumes or vapours, for example concentrated hydrochloric acid and ammonia, both of which can be thought of as saturated solutions of gases. The process of dilution is often highly exothermic (such that the increase in temperature can result in the boiling point of the solution being exceeded) so great care should be taken. Hazard information can be found in the CLEAPSS *Hazcards*¹, the *Hazardous Chemicals Manual CD2* (SSERC)² or in suppliers' Safety Data Sheets (SDS). **This information should be read and understood before any activity takes place.**

Model risk assessments

Preparing chemical solutions requires a risk assessment. This examines the hazards of using the chemicals and the proposed procedure. It then assesses the possibility of harm that could be experienced by the worker in order to decide which control measures should be employed to reduce the risk of harm. The control measures should be recorded. To save time, and as many of the control measures are the same no matter what substance is diluted, the control measures are recorded in model risk assessments.

Model risk assessments are available to those whose employers subscribe to CLEAPSS or SSERC (see below). Otherwise you should contact your employer for specific details. **This information should be read and understood before any activity takes place.**

In the methods given in section 2, the control measures are included in the written procedure, i.e. the text in daily use (the point-of-use text, as recommended by the DfEE³). This is the recommended approach as opposed to having tick boxes.

Inexperience and lone working

These are two particular hazards.



Those who have never prepared a dilute solution from a particularly hazardous concentrated solution or solid should seek advice and instruction from the senior science technician or an experienced teacher.



Some of these activities should not be carried out when working completely alone. There should be somebody who can come and assist should there be an accident.

CLEAPSS and SSERC also offer help over the telephone and provide courses where these issues are discussed (see below for contact details).

Moving chemicals

Another hazard is moving the chemical from the store to the bench.



If the chemical store entrance is in the prep room then the risk is slight. However, a spill kit should always be at hand to deal with an emergency. Eye protection should be worn.



If the store is not close at hand, carrying bottles of concentrated reagent from the store could cause a serious incident. Bottles of concentrated solutions should be carried in bottle carriers.



Movement of chemicals between laboratories and outside stores should **not** be performed at break times if many students are in corridors.

Emergencies

Technicians should be familiar with the immediate remedial measures to apply while waiting for first aid to arrive and also how to deal with other emergencies such as spills.

Further advice

More advice on hazards and risk assessments can be obtained from CLEAPSS and SSERC if your employer subscribes to one of these bodies.

In England, Wales and Northern Ireland:

CLEAPSS: www.cleapss.org.uk; email: science@cleapss.org.uk;
tel: 01895 251496

In Scotland:

SSERC: www.sserc.org.uk; email: sts@sserc.org.uk; tel: 01383 626070

¹ *Recipe Cards and Hazcards* (CLEAPSS, current edition, as on the annually updated CLEAPSS Science Publications CD-ROM).

² *Hazardous Chemicals Manual CD2* (SSERC, 2002, older versions on paper) contains many recipes and risk assessments.

³ *Safety in Science Education* (DfEE [now DCSF], 1996).

1.3 Calculations

Often, a calculation is needed to find the required mass of solid or liquid or the volume of liquid which is required to be diluted. However the guidance in section 1.4, and the recipes from CLEAPSS and SSERC, will cover many likely situations so you may be able to skip this section.

Volume

The SI unit for volume is m^3 , which represents a much larger volume (1000 litres¹) than is used in laboratories. It is more convenient to work in dm^3 and cm^3 . These are also called a litre and millilitre:

$$1 \text{ m}^3 \equiv 1000 \text{ dm}^3 \equiv 1\,000\,000 \text{ cm}^3 \equiv 1\,000\,000 \text{ ml}$$

$$1 \text{ litre} \equiv 1000 \text{ cm}^3 \equiv 1 \text{ dm}^3 \equiv 1000 \text{ ml}$$

Percentage concentrations

Concentrations are also quoted in different ways. The simplest method is to quote a percentage concentration:

w/v This represents the mass in grams of material per 100 cm^3 of solution.	w/w This represents the mass in grams of material per 100 g of solution.	v/v This represents the volume in cm^3 of material per 100 cm^3 of solution.
--	--	--

Molarity

Inconveniently, in chemical terms, a 1% (w/v) solution of sodium hydroxide contains a different number of sodium ions to a 1% (w/v) solution of sodium chloride. It is usual to quote concentration in terms of molarity so that, for example, 1 mol dm^{-3} sodium chloride and 1 mol dm^{-3} sodium hydroxide contain equal numbers of sodium ions.

If the formula mass² of a substance is dissolved in 1 dm^3 of solution, then that solution contains 1 mole dm^{-3} (abbreviation mol dm^{-3} or sometimes just M). The formula mass is usually quoted on the bottle or can often be found in catalogues.

The formula masses of sodium hydroxide and sodium chloride are 40 and 58.5 g mol^{-1} . So a 4% (w/v) sodium chloride solution and 5.85% (w/v) sodium hydroxide solution have the same concentration of sodium ions, although the percentages are different.

Concentration calculations

There are several methods of working out how much chemical to use, depending on whether you are dissolving a solid or a liquid or diluting down an existing solution.

Alternatively, use CLEAPSS and SSERC recipes or the table on page 9.

Dissolving a solid

$$1 \quad \text{mass of solid required} = (\text{molarity required}) \times (\text{volume required in dm}^3) \times \text{formula mass}$$

Example 1 Making 500 cm^3 solution of sodium hydroxide containing 0.4 mol dm^{-3} :
mass of solid sodium hydroxide required = $0.4 \times 0.5 \times 40 = 8 \text{ g}$

Dissolving a liquid

It is possible to use equation 1. However, if the density of the liquid is known it is often easier to measure it out by volume.

$$\mathbf{2} \text{ volume of liquid required} = \frac{(\text{molarity required}) \times (\text{volume required in dm}^3) \times \text{formula mass}}{\text{density of liquid}}$$

Example 2 Making 500 cm³ of sulfuric acid containing 1.5 mol dm⁻³. The density of concentrated sulfuric acid is 1.84 g cm⁻³, available from catalogues or data books.

$$\text{volume of concentrated sulfuric acid liquid required} = \frac{1.5 \times 0.5 \times 98}{1.84} = 39.9 \text{ cm}^3$$

Diluting a concentrated solution where the molarity is known

The following equation is useful for diluting solutions of known concentration.

$$\mathbf{3} \text{ volume of concentrated solution} = \frac{(\text{molarity of required solution}) \times (\text{volume of required solution})}{\text{molarity of concentrated solution}}$$

This equation can be written as:

$$V_1 = \frac{V_2 \times M_2}{M_1}$$

Example 3 Preparing 500 cm³ of 1.5 mol dm⁻³ hydrochloric acid from concentrated hydrochloric acid, approximately 12 mol dm⁻³ solution (see also *Example 8*):

$$M_1 = 12 \text{ mol dm}^{-3}; V_2 = 500 \text{ cm}^3; M_2 = 1.5 \text{ mol dm}^{-3}$$

$$V_1 = \frac{500 \times 1.5}{12} = 62.5 \text{ cm}^3$$

Example 4 Preparing 500 cm³ of 1.2 mol dm⁻³ hydrochloric acid from 2 mol dm⁻³ stock solution:

$$\text{volume of concentrated solution} = \frac{1.2 \times 500}{2} = 300 \text{ cm}^3$$

Example 5 Preparing 2.5 dm³ of 0.4 mol dm⁻³ sodium hydroxide from 2 mol dm⁻³ stock solution:

$$\text{volume of concentrated solution} = \frac{0.4 \times 2.5}{2} = 0.5 \text{ dm}^3$$

Example 6 Equation 3 can also be used where concentrations are expressed in units other than molarity, for example, hydrogen peroxide, which is often quoted as 'x vol (strength)'. This means one volume of the hydrogen peroxide solution will produce x volumes (measured in the same volume units) of oxygen at 0 °C and 1 atm pressure. So 1 cm³ of 20 vol hydrogen peroxide solution can produce 20 cm³ of oxygen.

Making 500 cm³ of 10 vol hydrogen peroxide from 100 vol solution:

$$\text{volume of concentrated solution} = \frac{10 \times 500}{100} = 50 \text{ cm}^3$$

Example 7 Making 500 cm³ of 1 mol dm⁻³ hydrogen peroxide from 100 vol solution: A 100 vol solution of hydrogen peroxide is approximately 8.8 mol dm⁻³

$$\text{volume of concentrated solution} = \frac{1 \times 500}{8.8} = 57 \text{ cm}^3$$

Diluting a concentrated solution where the %*(w/w)* is known

Some commercially available concentrated solutions, such as concentrated hydrochloric acid or '880' ammonia, are quoted in '%*(w/w)*'. You need to know the specific gravity of the concentrated solution; this is often quoted in catalogues or on Safety Data Sheets.

$$4 \quad \text{volume of concentrated solution} = \frac{(\text{concentration required}) \times (\text{volume in dm}^3) \times \text{formula mass} \times 100}{\% \text{ concentration of concentrated solution} \times \text{specific gravity}}$$

Example 8 Making 500 cm³ of 1.5 mol dm⁻³ hydrochloric acid (compare this with Example 3):

$$\text{volume of 36\% (w/w) hydrochloric acid} = \frac{1.5 \times 0.5 \times 36.5 \times 100}{36 \times 1.18} = 64.4 \text{ cm}^3$$

Diluting a concentrated solution to produce a required molarity

The following equation is useful for those confident with maths. It enables you to calculate the amount of water that needs to be added to a solution that is too concentrated. *Note:* this is suitable only where an approximate concentration is required because when water is mixed with a concentrated solution, the final volume of solution is not quite the sum of the two volumes.

$$5 \quad \text{Calculate: } x = \frac{\text{molarity of current solution}}{\text{molarity of required solution}}$$

For each 1 dm³ of concentrated solution add (x - 1 dm³) water.

Example 9 You have 1 dm³ of 2 mol dm⁻³ hydrochloric acid. The teacher wants 1.35 mol dm⁻³.

$$x = \frac{2}{1.35} = 1.48$$

So for 1 dm³ of the 2 mol dm⁻³ acid you need to add (1.48 - 1) or 0.48 dm³ of water.

¹Strictly speaking, 1 dm³ is defined differently from 1 litre but for most practical purposes – and certainly in the context of this booklet – the two can be taken as interchangeable.

²Sometimes the term 'relative formula mass' is used. This compares the formula mass of the chemical in question to that of hydrogen, which is 1 g mol⁻¹. Hence the relative formula mass is numerically the same as the formula mass, but has no units, as they cancel out.

1.4 Convenient recipes to make 1 dm³ of solution

If you find the use of arithmetic formulas difficult, you can use standard recipes listed in the table (or those published by CLEAPSS or SSERC) and adjust them accordingly. Note that these recipes will mostly produce stock solutions that will need to be further diluted for pupil use.

Standard recipes to prepare 1 dm³ (litre) of solution

0.1 mol dm ⁻³ aluminium potassium sulfate	47.4 g	of potassium aluminium sulfate-12-water
2 mol dm ⁻³ ammonia solution	115 cm ³	of '880' ammonia
0.1 mol dm ⁻³ barium chloride	24.4 g	of barium chloride-2-water
0.02 mol dm ⁻³ bromine water	1 cm ³	(1 small ampoule) of bromine liquid
0.1 mol dm ⁻³ copper(II) sulfate	25.0 g	of copper(II) sulfate-5-water
2 mol dm ⁻³ ethanoic acid	115 cm ³	of glacial ethanoic acid
2 mol dm ⁻³ hydrochloric acid	170 cm ³	of 36%(w/w) hydrochloric acid
20 vol hydrogen peroxide	200 cm ³	of 100 vol hydrogen peroxide
0.1 mol dm ⁻³ iodine solution	25.4 g	of iodine with 40 g of potassium iodide
0.1 mol dm ⁻³ iron(II) sulfate	27.8 g	of iron(II) sulfate-7-water to be dissolved in approx. 0.1 mol dm ⁻³ sulfuric acid, not water
0.1 mol dm ⁻³ iron(III) chloride	27.0 g	of iron(III) chloride-6-water to be dissolved in approx. 0.2 mol dm ⁻³ hydrochloric acid, not water
0.1 mol dm ⁻³ lead nitrate	33.1 g	of lead nitrate
2 mol dm ⁻³ nitric acid	124 cm ³	of 70% (w/v) nitric acid
2 mol dm ⁻³ phosphoric acid	116 cm ³	of 85% (w/v) phosphoric acid
0.1 mol dm ⁻³ potassium chloride	7.5 g	of potassium chloride
0.1 mol dm ⁻³ potassium iodide	16.6 g	of potassium iodide
0.1 mol dm ⁻³ potassium manganate(VII)	15.8 g	of potassium manganate(VII)
0.1 mol dm ⁻³ silver nitrate	17.0 g	of silver nitrate
1 mol dm ⁻³ sodium carbonate	286.1 g or 106 g	of sodium carbonate-10-water of sodium carbonate (anhydrous)
1 mol dm ⁻³ sodium hydrogencarbonate	84.0 g	of sodium hydrogencarbonate
2 mol dm ⁻³ sodium hydroxide	80.0 g	of sodium hydroxide
0.1 mol dm ⁻³ sodium thiosulfate	24.8 g	of sodium thiosulfate-5-water
1 mol dm ⁻³ sulfuric acid	54 cm ³	of concentrated sulfuric acid
0.1 mol dm ⁻³ zinc sulfate	28.8 g	of zinc sulfate-7-water

Example Making 500 cm³ of sulfuric acid containing 1.5 mol dm⁻³:
 1 dm³ of 1 mol dm⁻³ sulfuric acid is prepared from 54 cm³ concentrated acid.
 500 cm³ (0.5 dm³) of 1 mol dm⁻³ sulfuric acid is prepared from 54 × 0.5 concentrated acid, i.e. 27 cm³.
 500 cm³ of 1.5 mol dm⁻³ sulfuric acid is prepared from 27 × 1.5 cm³ concentrated acid, i.e. 40.5 cm³.

1.5 Accuracy

Solutions of approximate molarity can be used for qualitative work and sometimes even for quantitative work where accuracy is not critical. Do not be too worried about problems caused by rounding off figures in the arithmetic. The purity of the water, the assay of the chemical and precision of the equipment will also add to the errors. You will **not** cause major errors in the students' work with these calculations.

For some quantitative work the concentration or molarity of solutions needs to be accurately known. It is in the nature of the job to make solutions as accurately as possible. The solutions that need to be made with greatest care and accuracy are **standard** solutions. These are made with very pure chemicals. The mass used should be measured as accurately as possible and volumetric flasks should be used to measure the volume. Standard solutions are used as a reference against which the concentrations of other solutions are measured. Standard solutions are required when volumetric analysis is carried out.

It is important to make solutions up to the mark and not add together two known volumes of liquids and assume that they add accurately. There is often a small contraction in volume on mixing.

Examples The analysis of acids in vinegar is found by titration against sodium hydroxide solution. Solid sodium hydroxide absorbs water and is not easy to obtain pure. So the concentration of this prepared solution is only approximate and it will need to be standardised by titration against a standard acid solution of 0.1 mol dm^{-3} potassium hydrogen phthalate or of 0.1 mol dm^{-3} aminosulfonic acid (sulfamic acid). These two chemicals can be obtained in high purity and do not deteriorate on storage.

The analysis of carbonate in household washing soda is found by titration against hydrochloric acid solution. Concentrated hydrochloric acid loses hydrogen chloride on storage and its assay is not consistent. So its concentration is referenced to a standard alkali solution of 0.1 mol dm^{-3} sodium carbonate. This chemical can be bought in high purity and although it absorbs water on storage, this can be removed by warming.

Chemicals

The purity of the chemical is quoted in assay often printed on the label. It may also be printed in the catalogue and on the Safety Data Sheets (SDS). Only solids or solutions quoted as suitable for assay use, e.g. Analar, are guaranteed to be at least 99.5% pure. Labels on laboratory grade concentrated hydrochloric acid state that it is between 36 and 38% (w/w) pure. If a dilute solution is prepared from laboratory grade chemicals for volumetric work, it should be standardised against an assay grade chemical to determine its concentration.

Water

It is advisable to use pure water to make up solutions (distilled or deionised). Tap water contains anions that may cause cloudiness in some salt solutions and inaccuracies, especially in qualitative or titration work.

1.6 Equipment

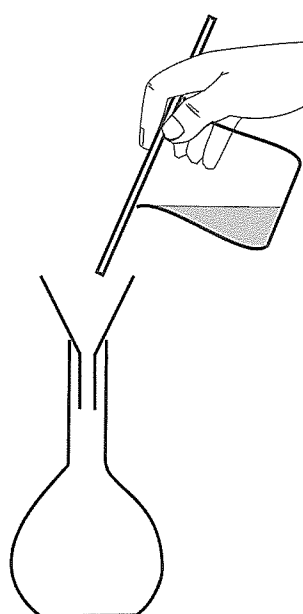
General points

Preparing large volumes of solutions presents manual handling problems as well as chemical. Although measuring cylinders to 500 cm³ capacity are generally suitable, physically handling and pouring from 1 or 2 dm³ tall-form measuring cylinders presents difficulty especially in a fume cupboard. 250 cm³ or even 100 cm³ cylinders are often adequate and much easier to use. A squat-form of measuring cylinder is available which is easier to handle although it often requires two hands to control it once the liquid is present.

A suitable alternative is a polypropylene laboratory measuring jug. The handle makes it ideal for handling solutions. A 2 dm³ jug is available but in some cases a technician may find that her or his wrist is not strong enough to cope with pouring 2 kg of liquid. CLEAPSS uses a jug on its technician course to make 2 mol dm⁻³ sulfuric acid, which is subsequently diluted and analysed. The results are surprisingly accurate so it is quite suitable for school chemistry. When using the jug, a magnetic stirrer is a useful device. The jug containing the magnetic stirrer bar can be placed on the stirrer. If solids are slow in dissolving, the technician can move to another job while waiting for the solid to dissolve. A combined heater-stirrer is a bonus but some materials change chemically on heating in water (e.g. iron(III) salts).

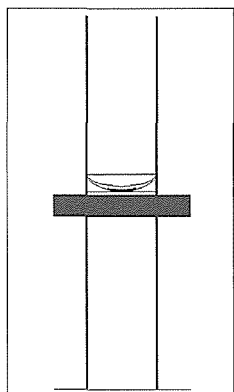
Another alternative is to use a 1 or 2.5 dm³ calibrated bottle with tape or waterproof labels stuck on the outside to mark the level for a specified volume. This has the advantage that liquid is less likely to be spilled. The bottle is placed in a sink on a piece of floor vinyl or similar material and filled with water equal in volume to approximately half the volume of the final solution. The required calculated volume of the concentrated acid, etc., is added slowly in portions with swirling and allowing time for heat to dissipate (chilled water can be used and further cooling effected by filling the sink with cold water). When all the acid, etc., is added, water is then added to make it up to the mark and the solution swirled again. Note no stirrer is used with this method. When swirling the bottle the cap should be loosely replaced to avoid spills. Solids can first be dissolved in a large beaker and the hotplate used as well if necessary. The concentrated solution can be decanted off into the calibrated bottle. As well as providing an additional source of cooling, the sink provides excellent containment in the event of an accident.

Wide-necked bottles are convenient for pouring and filling. Always label the bottles with the name of the chemical, the concentration and nature of hazard and the date of preparation.



Pouring down a glass rod

When it is important, in accurate work, to pour carefully from a beaker, etc., so that no drips are lost, experienced chemists pour down a glass rod. Place the rod across the top of the beaker, with the rod resting on the spout. Grasp the beaker in one hand, holding the rod in place with one finger. Tip the beaker and pour carefully and slowly. The solution should run along the rod and drops will form only at the end.



Reading a meniscus

To clarify the reference point of a meniscus, a strip of black paper may be placed round or just behind the tube; its reflection off the bottom of the meniscus provides a hard line at a reproducible, standardised reference point of the meniscus.

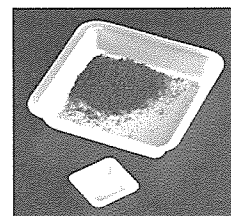
A trick used by experienced calibrators is to stick a strip of black electrical insulating tape on the bottom of a small mirror and hold this just below the meniscus, behind and in contact with the calibrated tube of the instrument. The reflection of the calibration line eliminates the parallax effect, while the black strip accentuates the reference point of the meniscus.

Examples of equipment used

Balance The number of decimal places to which the balance should measure mass depends on the level of work being carried out. A three-figure balance is required for some A-level work. A balance with a limit of 200 g may suffice if a very light weighing boat is used.



Weighing boat Never place chemicals directly on the balance pan. Very light plastic boats are available. No chemicals should be added to the boat while it is on the balance. This reduces the chance of a spill.



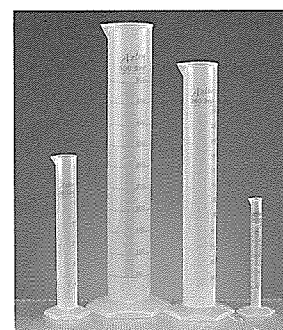
Spatula Use spatulas suitable for the mass to be measured. Always clean the spatula after each use.



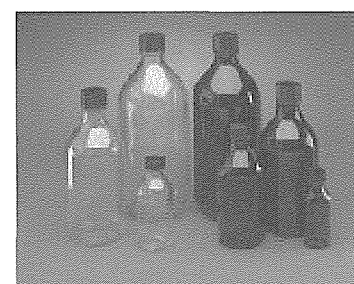
Magnetic stirrer These are available from about £60. You will also need to buy magnetic stirrer bars and a stirrer bar retriever is useful if the bar falls into the bottle or down the sink.



Measuring cylinder Use one suitable for the volume being measured. Never use a 100 cm³ cylinder to measure 40 cm³ of solution when a 50 cm³ measuring cylinder is available. Plastic measuring cylinders are just as accurate as glass apparatus.



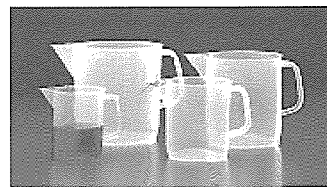
Storage bottles and labels Glass bottles are readily available; there are also plastic bottles that do not break easily. All bottles should be labelled with the name of the chemical, the concentration, the hazard and the date of preparation. Hazard labels can be printed from the *SSERC Hazardous chemicals manual CD* or from the *CLEAPSS Science publications CD-ROM*. For very large volumes an aspirator may be used – see below.



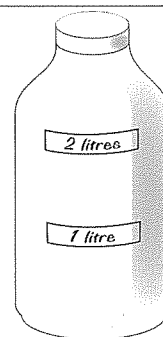
Laboratory jugs These are made of plastics (e.g. polypropylene), which withstand temperatures over 120 °C and hence can be put in the autoclave if required.

Their accuracy is often questioned but for making up solutions suitable for school use they are ideal. As long as the mass of solid or the volume of liquid are measured as accurately as possible then a difference of, for example, $\pm 20 \text{ cm}^3$ of solvent in 1000 cm^3 of solution represents a 2% error. For example, if the volume used in method 2.2 (page 17) had been 1.98 dm^3 rather than 2 dm^3 , then the concentration would have been $0.101 \text{ mol dm}^{-3}$. This would not cause a problem with titration work.

The handle means that they are easier to hold than a 2 dm^3 beaker. They are also easier to cope with than (tall) 2 or 1 dm^3 measuring cylinders when used in fume cupboards.

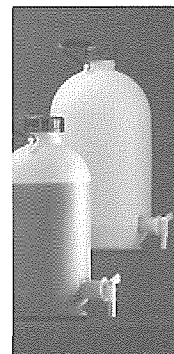


Calibrated bottles 1 or 2.5 dm^3 calibrated bottles, with tape or waterproof labels stuck on the outside to mark the level for a specified volume, can be used instead of jugs. These can hold stated volumes with more than sufficient accuracy for working solutions. As they can be swirled safely (with the cap in place) for mixing solutions, a magnetic stirrer is not needed.



Aspirator It is tempting to buy aspirators with a capacity of 10 dm^3 or more but remember that they may have to be moved. A 5 dm^3 aspirator would usually contain 5 kg of solution (if the density of the liquid is 1 kg dm^{-3}). Unless trolleys are used to convey aspirators, this is a convenient load to carry by hand a distance of no more than 10 m. Keep the taps clean, especially those holding sodium hydroxide solutions.

Care should be taken if the aspirator contains anything other than water when it is being moved. If alkalis, such as NaOH, are being stored, then glass taps should not be used as they can seize up. Buy one that has spare taps available and buy the spare when you buy the original. Add a drip tray underneath the tap or hang a small 'beaker' from it of about 250 cm^3 capacity, cut from a plastic bottle, to catch any drips.

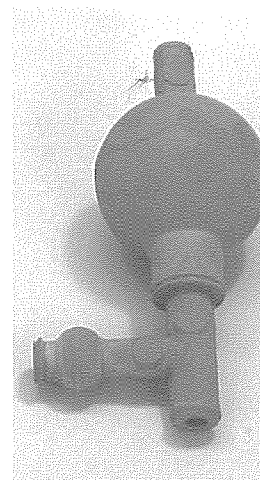


Bulb pipette Bulb (single mark) pipettes are designed for the precise delivery of single quantities of liquid. They have a very narrow 'mouthpiece tube' widening into a large capacity bulb before narrowing again into another narrow tube terminating in a jet. The bulb enables larger capacity pipettes to be of manageable short length for easy handling. The narrow mouthpiece tube carries the calibration ring to give sensitivity for accurate filling. The narrow delivery tube is suitable for insertion into narrow-necked vessels, such as flasks, for sampling.

Safety note: Although it has a 'mouthpiece tube', the pipette should never be put in the mouth. Always use a safety filler.



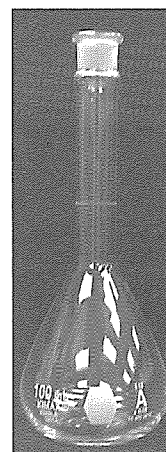
Pipette safety fillers As there is no pipetting by mouth nowadays, these are very important. The rubber bulb pipettes (illustrated) must be kept free of liquid and never put away 'squeezed'. Alkalis react with rubber and continual contamination with alkalis renders the bulb useless, so other types are worth considering. The ratchet-type design depends on the fitting being as tight as possible. It needs time and patience to use a filler correctly. Automatic ones are very expensive. The less expensive 10 cm³ automatic pipettor, available from suppliers for about £20, produces reproducible results but requires calibration before use.



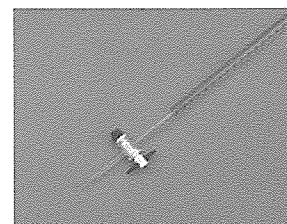
Volumetric flasks The volumetric flask is a primary tool of an analyst in that it has a large-capacity bulbous bottom and long narrow neck with a single calibration ring to allow for accurate filling. Designed to contain their stated capacity, these flasks are frequently used to prepare 'standard solutions', the basic reference reagents for a range of analytical methods.

The most common error is not to mix the solution well prior to use. Once the stopper has been fitted, invert the flask about ten times to ensure thorough mixing. A finger or thumb must be used to hold the stopper firmly in place when the flask is inverted.

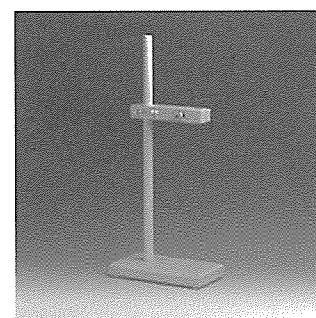
Note that for most salts, acids, etc., a reduction in volume occurs when water or a dilute solution (as in the neck of the flask) mixes with a more concentrated one (in the body of the flask). Therefore, once the contents have been well mixed check again that the level of the solution is up to the mark and top up if necessary.



Burette The burette enables the chemist to add a precise volume of a reagent to a known volume of another reagent. An indicator is used to monitor when one reagent just reacts with all the other reagents. (More sophisticated methods, such as pH probes, may be used instead.) It is usual to place the acid in the burette and the alkali in a conical flask because alkalis can affect the glass in the burette and, after years of use, this renders the burette less accurate. Alkalis also react with the grease used to lubricate the glass tap on the burette. If schools are using white PTFE taps, this is not so important. When reading the level, pick up the burette in its stand and place it on a stool so that your eye can be exactly level with the meniscus. Consider buying 25 cm³ burettes rather than 50 cm³. The smaller burette is not as tall and it is much easier to read the zero level without having to lift the equipment to a lower level. However, it may not be suitable for all purposes, nor meet the requirements of some examination boards.



Burette stand It is advisable to use a wooden burette stand with the provided clamp. The problem with normal metal clamps is that the clamp may be turned so hard that it breaks the glass burette. There are special burette holders that can be attached to metal retort stands. However, those designed for a 50 cm³ burette may not always fit a 25 cm³ burette.



2 Techniques for preparing various solutions

2.1 Preparing 500 cm³ of approximately 0.1 mol dm⁻³ copper sulfate solution



Hazards

Copper sulfate solution is **HARMFUL** if swallowed. Solutions equal to or stronger than 1 mol dm⁻³ should be labelled **HARMFUL**. It may also be irritating to the eyes and skin and has been known to sensitise the skin. It is very **TOXIC** to the aquatic environment and may cause long-term adverse effects.

Chemicals required

12.5 g of copper(II) sulfate-5-water (**HARMFUL**), pure water

Equipment required

Weighing boat, balance, spatula, 600 cm³ beaker, magnetic stirrer and stirrer bar, wash bottle, 500 cm³ measuring cylinder [OR 1 dm³ calibrated bottle], storage bottle, funnel

Procedure with control measures

- 1 Wear eye protection, gloves and apron or labcoat.
- 2 Place the weighing boat on a balance and tare the reading.
- 3 Measure out with a spatula 12.5 g of copper(II) sulfate-5-water (**HARMFUL**) on the weighing boat.
- 4 **Do not** return any unused salt to the bottle. Wipe the bottle. Return the bottle to its storage place.
- 5 Pour about 300 cm³ of pure water into a 600 cm³ beaker. Place this on a magnetic stirrer and add a stirrer bar.
- 6 Add the copper(II) sulfate to the beaker containing water. Rinse the weighing boat with water from a wash bottle and add the washings to the beaker.
- 7 Start the stirrer.
- 8 When the salt has dissolved, pour the solution into a 500 cm³ measuring cylinder [or a calibrated bottle].
- 9 Rinse the beaker out with three portions of water and add the washings to the measuring cylinder [or a calibrated bottle].
- 10 *Measuring cylinder method.* Pour the solution back into the beaker a couple of times to ensure good mixing.
Calibrated bottle method. Put the cap on the calibrated bottle and swirl thoroughly.
- 11 Add water to the measuring cylinder [or calibrated bottle] so that the level reads '500 cm³'.
- 12 Pour the solution into an appropriately labelled bottle using a funnel.
- 13 Clean and rinse all the equipment.

2.2 Preparing 2 dm³ of approximately 0.1 mol dm⁻³ sodium thiosulfate solution

**Hazards**

Sodium thiosulfate has minimal hazards but would be HARMFUL if ingested in quantity.

Chemicals required

49.64 g of sodium thiosulfate-5-water, chilled pure water

Equipment required

2 dm³ jug (OR two 1 dm³ jugs) [OR 2.5 dm³ calibrated bottle and large beaker], spatula, large weighing boat or small beaker, balance, magnetic stirrer and stirrer bar, wash bottle, storage bottle, funnel

Procedure with control measures

- 1 Wear eye protection. If you find a 2 dm³ jug too heavy to handle, then use two 1 dm³ jugs or a 2.5 dm³ calibrated bottle and adjust the method accordingly.
- 2 Place the weighing boat or beaker on a balance and tare the reading.
- 3 Measure out with a spatula 49.64 g of sodium thiosulfate-5-water on the large weighing boat or in the beaker.
- 4 **Do not** return any unused salt to the bottle. Wipe the bottle. Replace the screw top. Return the bottle to its storage place.
- 5 *Jug method.* Place a magnetic stirrer bar in a 2 dm³ measuring jug and fill the jug half-full with pure water. Place this on a magnetic stirrer. Start the stirrer.
Calibrated bottle method. One third fill a calibrated bottle with chilled pure water and place in sink.
- 6 *Jug method.* Add the sodium thiosulfate-5-water to the jug containing water.
Calibrated bottle method. Dissolve the sodium thiosulfate-5-water in about 600 cm³ pure water in a large beaker on a stirrer hotplate.
- 7 Rinse the weighing boat or small beaker with water from a wash bottle and add the washings to the jug [or large beaker].
- 8 When the solid has dissolved, stop the stirrer.
- 9 *Jug method.* Add pure water so that the level reaches the 2 dm³ mark. Restart the stirrer to ensure thorough mixing.
Calibrated bottle method. Transfer the solution to the calibrated bottle and make up to the mark with pure water. Swirl to ensure thorough mixing.
- 10 Pour the solution into an appropriately labelled bottle using a funnel.
- 11 Clean and rinse all the equipment.

2.3 Preparing 2 dm³ of approximately 2 mol dm⁻³ sodium hydroxide solution



Hazards

Sodium hydroxide pellets and solutions of 0.5 mol dm⁻³ or greater concentration are **CORROSIVE**. Solutions of 0.05 mol dm⁻³ but less than 0.5 mol dm⁻³ are **IRRITANT**. Wear eye protection to BS EN166 3 standard (goggles with no vents or indirect vents) and an apron or labcoat.

Chemistry

Sodium hydroxide pellets absorb water and carbon dioxide from the atmosphere. Bottles should be securely closed after use. It is inadvisable to use pearls of sodium hydroxide as they can cake together and be hazardous.

Pellets also cake together (but less than 'pearls') and separation is only possible by physically separating them with a screwdriver or similar. Wear eye protection to BS EN 166 3 standard. Make sure the solid is contained so that pellets do not shoot out onto the bench or floor.

Sodium hydroxide pellets and solutions are continually absorbing carbon dioxide and accurate concentrations (especially of dilute solutions) cannot be guaranteed. For accurate titration work, the prepared solution should be standardised against standard potassium hydrogen phthalate solution immediately before use.

If more concentrated solutions are prepared, several methods can be used. The solutions can be cooled in an ice-bath mixture between additions of the solid hydroxide. Or the solution can be made by dissolving the first portion of sodium hydroxide pellets in a mixture of pure water and ice made from pure water. Finally, extra solid sodium hydroxide can be added to a 2 mol dm⁻³ sodium hydroxide solution rather than starting with pure water.

Chemicals required

160 g of sodium hydroxide pellets

Equipment required

2 dm³ jugs (OR two 1 dm³ jugs) [OR 2.5 dm³ calibrated bottle and large beaker], spatula, small beaker (a weighing boat will be too small), balance, magnetic stirrer and stirrer bar, wash bottle, aspirator, funnel

Procedure with control measures

- 1 Wear eye protection to BS EN 166 3 standard. If you find a 2 dm³ jug too heavy to handle, use two 1 dm³ jugs or a 2.5 dm³ calibrated bottle and adjust the method accordingly.
- 2 Place the small beaker on a balance and tare the reading.
- 3 Measure out with a spatula 160 g of sodium hydroxide pellets.
- 4 It is not advisable to return any unused solid to the bottle; take care not to dispense too much. Wipe the bottle. Replace the screw top. Return the bottle to its storage place.
- 5 *Jug method.* Place a magnetic stirrer bar in a 2 dm³ measuring jug and fill the jug three-quarters-full with pure water. Place this on a magnetic stirrer. *Calibrated bottle method.* Place a magnetic stirrer bar in a large beaker and fill three-quarters-full with pure water. Place this on a magnetic stirrer.
- 6 Start the stirrer.
- 7 Add a quarter of the sodium hydroxide pellets to the jug [or large beaker] containing water (this is best done after starting the stirrer to lower the risk of the pellets clumping together).
- 8 Stop the stirrer to check the solid has dissolved. Start the stirrer and add a further quarter of the sodium hydroxide pellets.
- 9 Repeat the dissolving until all the sodium hydroxide has been added.
- 10 Rinse the small beaker which contained the pellets with water and add the washings to the jug [or large beaker].
- 11 Stop the stirrer. This is a very exothermic reaction, so the solution should be allowed to cool at this stage before the final dilution is carried out.
- 12 *Jug method.* Add pure water so that the level reaches the 2 dm³ mark. Restart the stirrer to ensure thorough mixing. *Calibrated bottle method.* Transfer the solution to the calibrated bottle, standing in a sink, and make up to the mark with pure water. Swirl to ensure thorough mixing.
- 13 Pour the solution into an appropriately labelled aspirator using a funnel.
- 14 Clean and rinse all the equipment.

2.4 Preparing 500 cm³ of approximately 0.4 mol dm⁻³ solution for general laboratory work from a prepared 2 mol dm⁻³ stock solution



Hazards Wear eye protection. If the solution you are diluting is labelled CORROSIVE or TOXIC, then wear eye protection to BS EN 166 3 standard (goggles with no vent or indirect vent), an apron or labcoat and, if relevant, appropriate gloves for the particular chemical (see *Hazardous chemicals manual CD2* or *Hazcards*).

Chemistry Using equation 3:

$$\text{volume of concentrated solution} = \frac{0.4 \times 500}{2} \text{ or } 100 \text{ cm}^3$$

Chemicals required 100 cm³ of 2 mol dm⁻³ solution, pure water

Equipment required 100 cm³ measuring cylinder, 500 cm³ measuring cylinder and large beaker [OR 1 dm³ calibrated bottle], storage bottle, funnel

- Procedure with control measures**
- 1 Wear the necessary eye protection.
 - 2 Measure 100 cm³ of the 2 mol dm⁻³ solution in a 100 cm³ measuring cylinder.
 - 3 *Measuring cylinder method.* Fill a 500 cm³ measuring cylinder half-full with pure water. Add the 100 cm³ of the 2 mol dm⁻³ solution from the measuring cylinder to the water.
Calibrated bottle method. Pour about 250 cm³ of chilled pure water into a calibrated bottle and place in sink. Add the 100 cm³ of the 2 mol dm⁻³ solution from the measuring cylinder and swirl.
 - 4 *Measuring cylinder method.* Add more water so that it reaches the 500 cm³ mark. Pour backwards and forwards into a large beaker to mix thoroughly.
Calibrated bottle method. Add more water so that it reaches the 500 cm³ mark and swirl to mix.
 - 5 Pour the solution into an appropriately labelled bottle using a funnel.
 - 6 Clean and rinse all the equipment.

2.5 Preparing 1 dm³ of a 0.1 mol dm⁻³ standard solution for volumetric work from an exactly 2 mol dm⁻³ solution



Hazards

Wear eye protection. If the solution you are diluting is labelled CORROSIVE or TOXIC, wear eye protection to BS EN 166 3 standard (goggles with no vent or indirect vent), an apron or labcoat and, if relevant, appropriate gloves for the particular chemical (see *Hazardous chemicals manual CD2* or *Hazcards*).

Chemistry

Using equation 3:

$$\text{volume of concentrated solution} = \frac{0.1 \times 1000}{2} \text{ or } 50 \text{ cm}^3$$

Chemicals required

75 cm³ of exactly 2 mol dm⁻³ solution, pure water

Equipment required

1 dm³ volumetric flask, funnel, 25 cm³ pipette, pipette safety filler, wash bottle with fine tip

Procedure with control measures

- 1 Wear the necessary eye protection.
- 2 Using a funnel, add about 500 cm³ of pure water to the 1 dm³ volumetric flask.
- 3 Attach the pipette safety filler to the 25 cm³ pipette.
- 4 Withdraw about 25 cm³ of the 2 mol dm⁻³ solution and expel the solution into a waste container. This procedure rinses out the pipette.
- 5 Withdraw over 25 cm³ of the 2 mol dm⁻³ solution into the pipette and carefully adjust it at eye level to the designated mark. Make sure there are no drops on the outside of the pipette and then allow the liquid to run into the volumetric flask. Once the liquid has run into the flask, touch the tip against the inside of the neck of the flask so that another small drop is delivered. **Do not** blow out the small amount of liquid remaining in the tip of the pipette.
- 6 Repeat the procedure so that 50 cm³ of liquid has been added.
- 7 Using the funnel, add more water to the volumetric flask until the liquid level is just below the designated 1000 cm³ mark. Remove the funnel.
- 8 Now, using a wash bottle with a fine tip, add water carefully until the correct amount has been added. Insert the stopper.
- 9 Invert the volumetric flask 10 times to ensure thorough mixing. Do not be concerned if the final level is slightly below the mark as some liquid might be adhering to the stopper.
- 10 Label the flask.
- 11 Clean and rinse all the equipment.
- 12 Heat is often generated during this procedure, so it is advisable to do the dilution a day in advance and check the level on the morning of use, topping up if necessary and then inverting and swirling again.

2.6 Preparing 2 dm³ of approximately 2 mol dm⁻³ hydrochloric acid



Hazards	Concentrated hydrochloric acid is CORROSIVE . Eye protection to BS EN 166 3 standard should be worn (goggles with no vent or indirect vent). Nitrile gloves that protect against chemical splash should be worn. The hydrogen chloride vapour is TOXIC so this operation must be carried out in a working fume cupboard. Handling 1 or 2 dm ³ measuring cylinders in a fume cupboard is awkward so a measuring jug or calibrated 2.5 dm ³ bottle is recommended.
Chemistry	Concentrated hydrochloric acid is sold in different formulations: the most common is 35–38% (w/w). The assay is variable and gas is lost during storage once the bottle has been opened. If hydrochloric acid is required for accurate analysis work, it needs to be standardised against sodium carbonate solution.
Chemicals required	360 cm ³ of concentrated hydrochloric acid, chilled pure water
Equipment required	2 dm ³ jug (OR two 1 dm ³ jugs) [OR 2.5 dm ³ calibrated bottle], 500 cm ³ reagent bottle, 250 cm ³ measuring cylinder, magnetic stirrer and stirrer bar (only for the jug method), storage bottle, funnel
Procedure with control measures	<ol style="list-style-type: none"> 1 Wear eye protection to BS EN 166 3 standard (goggles with no vent or indirect vent). If you find a 2 dm³ jug too heavy to handle, use two 1 dm³ jugs and adjust the method accordingly. Wear nitrile gloves. Carry out this procedure in a working fume cupboard. 2 Pour about 360 cm³ of concentrated hydrochloric acid into a 500 cm³ reagent bottle. (This avoids the difficult operation of pouring the acid from a heavy 2.5 dm³ bottle into a measuring cylinder.) From this, measure 170 cm³ of acid in a 250 cm³ measuring cylinder. 3 <i>Jug method.</i> Place a magnetic stirrer bar in a 2 dm³ measuring jug and fill the jug half-full with pure water. Place this on a magnetic stirrer. Start the stirrer. <i>Calibrated bottle method.</i> Place a 2.5 dm³ calibrated bottle in the sink and pour in about 1 dm³ of chilled water. 4 A little at a time, add the 170 cm³ of the acid to the jug [or calibrated bottle] containing water. 5 Measure out a further 170 cm³ of the concentrated hydrochloric acid. Again, a little at a time, add this to the jug [or calibrated bottle]. 6 Do not return any unused acid from the reagent bottle to the main stock bottle in case it has become contaminated. Wipe the bottle, including the neck. Replace the screw top. Return the bottle to its storage place. 7 Fill the measuring cylinder with water and pour this into the jug [or calibrated bottle]. 8 Repeat the last instruction. 9 <i>Jug method.</i> Stop the stirrer, wait until the solution has reached room temperature, and then add water until the required volume of solution is reached. Restart the stirrer to ensure thorough mixing. <i>Calibrated bottle method.</i> Swirl the contents, wait until the solution has reached room temperature, and then add water until the required volume of solution is reached. Swirl again to ensure thorough mixing. 10 Pour the solution into an appropriately labelled bottle using a funnel. 11 Clean and rinse all the equipment.

2.7 Preparing 2 dm³ of approximately 2 mol dm⁻³ ammonia solution



Hazards

Concentrated ammonia is **CORROSIVE**. It continually produces ammonia gas which is **TOXIC**. All work must be carried out in a working fume cupboard. Eye protection to BS EN 166 3 standard should be worn. Nitrile gloves that protect against chemical splash should be worn. Handling 1 or 2 dm³ measuring cylinders in a fume cupboard is awkward so a measuring jug or calibrated 2.5 dm³ bottle is recommended.

Chemistry

Concentrated ammonia is sold in different formulations: the most common is known as '880' (specific gravity 0.88). The assay is variable and gas is lost during storage once the bottle has been opened. If ammonia is required for accurate analysis work, it needs to be standardised against hydrochloric acid* that has been standardised against sodium carbonate solution.

Chemicals required

230 cm³ of ammonia, chilled pure water

Equipment required

2 dm³ jug (OR two 1 dm³ jugs) [OR 2.5 dm³ calibrated bottle], 250 cm³ reagent bottle, 250 cm³ measuring cylinder, magnetic stirrer and stirrer bar (only for the jug method), storage bottle, funnel

Procedure with control measures

- 1 Wear eye protection to BS EN 166 3 standard, nitrile gloves and an apron or labcoat. If you find a 2 dm³ jug too heavy to handle, use two 1 dm³ jugs or a 2.5 dm³ calibrated bottle and adjust the method accordingly. Do this procedure in a working fume cupboard.
- 2 **Take care** when opening the ammonia bottle especially in hot weather. Pour about 250 cm³ of concentrated ammonia into a 250 cm³ reagent bottle. (This avoids the difficult operation of pouring the ammonia from a heavy 2.5 dm³ bottle into a measuring cylinder.)
- 3 Measure 230 cm³ of ammonia from the reagent bottle into a 250 cm³ measuring cylinder.
- 4 **Do not** return any unused ammonia to the main stock bottle in case it has become contaminated. Wipe the bottle, including the neck. Replace the screw top. Return the bottle to its storage place.
- 6 *Jug method.* Place a magnetic stirrer bar in a 2 dm³ measuring jug and fill the jug half-full with pure water. Place this on a magnetic stirrer. Start the stirrer.
Calibrated bottle method. Place a 2.5 dm³ calibrated bottle in the sink and pour in about 1 dm³ of chilled water.
- 7 Add the 230 cm³ of ammonia to the jug [or calibrated bottle] containing water.
- 8 Fill the measuring cylinder with water and pour this into the jug [or calibrated bottle].
- 9 Repeat the last instruction.
- 10 *Jug method.* Stop the stirrer and then add water until the required volume of solution is reached. Restart the stirrer to ensure thorough mixing.
Calibrated bottle method. Swirl the contents and then add water until the required volume of solution is reached. Swirl again to ensure thorough mixing.
- 11 Pour the solution into an appropriately labelled bottle using a funnel.
- 12 Clean and rinse all the equipment.

*Aminosulfonic acid (sulfamic acid) is a good primary standard because it can be weighed out directly and does not need standardisation. It is a strong acid and therefore suitable for titration against weak bases such as ammonia as well as strong bases such as sodium hydroxide.

2.8 Preparing 2 dm³ of approximately 1 mol dm⁻³ sulfuric acid



Hazards

Concentrated sulfuric acid is **CORROSIVE**, causing serious burns. The energy released when concentrated sulfuric acid is added to water is considerable. The temperature can rise so rapidly that the solution boils and hot acid is sprayed out of the container, causing serious burns. **So acid is always added to water** and this must be done **slowly with good mixing**. You should **not** attempt this procedure for the first time on your own. A more experienced technician or teacher should supervise what you do.

Apparatus used to hold concentrated sulfuric acid **must be dry** before use.

Chemistry

The use of an ice/water mixture, in place of water at room temperature, makes this procedure safer. The diluted solution produced is already at about room temperature and ready for use. The control measure makes use of the heat taken in when ice melts. The method is suitable for making bulk solutions of 1 or 2 mol dm⁻³ sulfuric acid. It is suggested that 1 dm³ at a time is made up and the diluted solution is added to labelled bottles.

Chemicals required

108 cm³ concentrated sulfuric acid, pure water and ice made from pure water

Equipment required

100 or 250 cm³ reagent bottle, 100 cm³ measuring cylinder, 1 dm³ measuring jug [OR calibrated bottle], magnetic stirrer and stirrer bar (only for the jug method), storage bottle, funnel

Procedure with control measures

To make 1 dm³ of dilute sulfuric acid

- 1 Wear a face shield and a laboratory coat. Wear nitrile gloves and a PVC apron which protect from chemical splash.
- 2 Pour about 60–70 cm³ of the concentrated sulfuric acid from the stock bottle into a 100 or 250 cm³ **dry** reagent bottle. (This avoids the difficult operation of pouring the acid from a heavy 2.5 dm³ bottle into a measuring cylinder.)
- 3 Measure 54 cm³ of the concentrated sulfuric acid from the reagent bottle into a **dry** 100 cm³ measuring cylinder.
- 4 **Do not** pour any unused concentrated acid back into the main stock bottle because it may have become contaminated.
- 5 *Jug method.* Place a magnetic stirrer bar into a 1 dm³ measuring jug followed by 50 cm³ of pure water and then ice (made from pure water) so that the jug is approximately two-thirds full. Place the jug on a magnetic stirrer.

Calibrated bottle method. Place the 1 dm³ calibrated bottle in a sink on a small piece of vinyl flooring material and half-fill with ice-chilled water.

- 6 *Jug method.* Start the stirrer. Gently pour about a third of the volume of the acid from the measuring cylinder onto the ice in the measuring jug. Ice sometimes stops the magnetic stirrer from turning so some initial help may be required with a glass rod. However, stir gently as the glass stirrer might break or cause splashing.

Calibrated bottle method. Gently pour about a third of the volume of the acid from the measuring cylinder into the ice-chilled water in the calibrated bottle. Swirl carefully with the cap in place.

- 7 Repeat with a second and third portion of acid and keep stirring [or swirling]. The ice in the jug should now have melted.
- 8 *Jug method.* Stop the stirrer. When the solution has reached room temperature add pure water to the jug until the required volume is reached. Restart the stirrer to ensure thorough mixing.

Calibrated bottle method. When the solution has reached room temperature add pure water to the calibrated bottle until the required volume is reached. Swirl to ensure thorough mixing.

- 9 Pour the solution via a funnel into an appropriately labelled bottle.

To make 2 dm³ of dilute sulfuric acid

- 10 Repeat steps 1 to 9, adding the newly diluted acid to the same stock bottle in step 9.
- 11 Clean and rinse all the equipment.

2.9 Preparing 250 cm³ of a 0.1 mol dm⁻³ standard solution of sodium carbonate



Hazards

Sodium carbonate is an IRRITANT. Wear eye protection. When using tongs to move hot objects, ensure the tongs meet correctly.

Chemistry

This alkaline solution can be made to a high standard of accuracy. The solution needs to be made as carefully and accurately as possible so sufficient time needs to be allowed. High-purity grade **anhydrous** sodium carbonate is required. It absorbs water and carbon dioxide from the atmosphere and needs heating before using. If heated to too high a temperature, sodium carbonate does decompose, contrary to what is stated in some textbooks. Pure water must be used.* Do not leave bottles of this solution open to the atmosphere as carbon dioxide is absorbed. It should be made just before use and not left for use several weeks later.

Chemicals required

About 3 g high-purity grade anhydrous sodium carbonate (Analar or equivalent), pure water

Equipment required

Evaporating basin or beaker, oven, tongs, desiccator, 100 cm³ beaker, balance (use the most accurate available in the lab), spatula, glass stirring rod, funnel, 250 cm³ volumetric flask, wash bottle

Procedure with control measures

- 1 Place about 3 g of high-purity sodium carbonate in an evaporating basin (or beaker) and warm it in an oven (250 °C for 30 minutes).
- 2 Using the tongs, transfer the hot evaporating basin (or beaker) to a desiccator and let it cool (**care: hot object**). The desiccator must have a release tap to open once it is cool, or be very slightly ajar while it cools, or the lid will become impossible to remove.
- 3 Place a 100 cm³ beaker on the pan of the balance and tare the reading.
- 4 Using the spatula, weigh out accurately 2.650 g of the anhydrous sodium carbonate into the beaker.
- 5 Now add about 50 cm³ of pure water and stir carefully with the glass stirring rod to avoid splashing until the solid dissolves.
- 6 Pour the solution carefully down the glass stirring rod (see page 12), through the funnel into the volumetric flask.
- 7 Add about 25 cm³ of pure water to the beaker, stir it and add the water to the volumetric flask.
- 8 Repeat the previous instruction.
- 9 Using the wash bottle, rinse the stirring rod, with the washings going into the volumetric flask.
- 10 Using the wash bottle, rinse around the funnel, with the washings going into the volumetric flask.
- 11 Add pure water from a beaker to the volumetric flask and when the level is at the base of the neck, with the meniscus at eye level, add the water dropwise until the bottom of the meniscus is level with the graduation mark.
- 12 Insert the stopper and, holding it in place firmly, invert the flask several times and swirl to make sure the solution is mixed properly. If necessary make up to the mark again.
- 13 Label the volumetric flask.
- 14 Wash all equipment.

*Another substance that can be obtained in a very pure state and can be used as a standard is sodium tetraborate-10-water (borax), as it does not require this initial heating. The chemistry may be considered rather involved for students.

2.10 Preparing 250 cm³ of a 0.1 mol dm⁻³ standard solution of potassium hydrogen phthalate



Hazards

When using tongs to move hot objects, ensure the tongs meet correctly. The pH of this solution is 4.

Chemistry

This acidic solution can be made to a high standard of accuracy. The solution needs to be made as carefully and accurately as possible so sufficient time needs to be allowed. High-purity grade potassium hydrogen phthalate is required.* It absorbs a little water from the atmosphere and needs warming before using. If heated to too high a temperature, potassium hydrogen phthalate may decompose. Pure water must be used.

Chemicals required

About 6 g high-purity grade potassium hydrogen phthalate, pure water

Equipment required

Evaporating basin or beaker, oven, tongs, desiccator, 100 cm³ beaker, balance (use the most accurate available in the lab), spatula, glass stirring rod, funnel, 250 cm³ volumetric flask, wash bottle

Procedure with control measures

- 1 Place about 6 g of high-purity potassium hydrogen phthalate in an evaporating basin (or beaker) and warm it in an oven (120 °C for 30 minutes).
- 2 Using the tongs, transfer the hot evaporating basin (or beaker) to a desiccator and let it cool (**care: hot object**).
- 3 Place the 100 cm³ beaker on the pan of the balance and tare the reading.
- 4 Using the spatula, weigh out accurately 5.101 g of anhydrous potassium hydrogen phthalate into the beaker.
- 5 Boil some pure water (**care: hot object**).
- 6 Now pour about 50 cm³ of the boiled water onto the weighed potassium hydrogen phthalate in the beaker. Stir carefully with the glass stirring rod to avoid splashing until the solid dissolves.
- 7 Add a little cold pure water to cool the solution and pour the solution carefully down the glass stirring rod (see page 12), through the funnel into the volumetric flask.
- 8 Add about 25 cm³ of pure water to the beaker, stir it and add the water to the volumetric flask.
- 9 Repeat the previous instruction.
- 10 Using the wash bottle, rinse the stirring rod, with the washings going into the volumetric flask.
- 11 Using the wash bottle, rinse around the funnel, with the washings going into the volumetric flask.
- 12 Add pure water from a beaker to the volumetric flask and when the level is at the base of the neck, with the meniscus at eye level, add the water dropwise until the bottom of the meniscus is level with the graduation mark.
- 13 Insert the stopper and, holding it in place firmly, invert the flask several times and swirl to make sure the solution is mixed properly. If necessary make up to the mark again.
- 14 Label the volumetric flask.
- 15 Wash all equipment.

*Using this material may seem too elaborate for school use. As an alternative, dilute hydrochloric acid, which has been standardised against sodium carbonate solution, can be used as an acidic solution whose concentration is known accurately.

2.11 Preparing 100 cm³ of 1% (w/v) 'starch solution'

**Hazards**

The liquids and beaker will be hot. Wear eye protection. Thermal gloves or other means of picking up hot glassware and pouring the liquids will be needed.

Chemistry

Starch is a polysaccharide made up of hundreds of glucose units chemically bonded together. It is called a polymer. It does not 'dissolve' in water as such. The solution we refer to as 'starch solution' is a colloidal suspension. Hopefully, the long strands of starch units have been teased apart by the addition of boiling water and remain suspended. In time, the units 'tangle up' again and solid starch precipitates out. The 'solution' should be made the day before it is required so that the temperature of the solution has time to equilibrate with the surroundings. If there is some settlement of starch from the solution, it should not make much difference to it being used.

Chemicals required

1 g starch, **small** pinch of sodium benzoate or benzoic acid (optional), pure water

Equipment required

Kettle or beaker for heating water, Bunsen burner, tripod and gauze, 250 cm³ beaker, glass stirrer, 100 cm³ measuring cylinder

Procedure with control measures

- 1 Wear eye protection.
- 2 Boil some pure water in a kettle or about 70 cm³ in a 250 cm³ beaker.
- 3 Measure out 1 g of starch and add this to a 250 cm³ beaker.
- 4 Add a little pure water and mix it well into a paste with a glass stirrer.
- 5 Add about 70 cm³ of boiling water and continue to stir.
- 6 (**Care: the beaker is hot**) Pour this solution into a 100 cm³ measuring cylinder and make up to the mark with pure water.
- 7 Pour the solution back into the 250 cm³ beaker.
- 8 Place the beaker on a tripod and gauze and bring the liquid to boiling point with the hot flame of a Bunsen burner. A **small** pinch of sodium benzoate or benzoic acid can be added to aid its keeping properties.
- 9 (**Care: the beaker is hot**) Allow to cool and pour this solution into a labelled bottle.

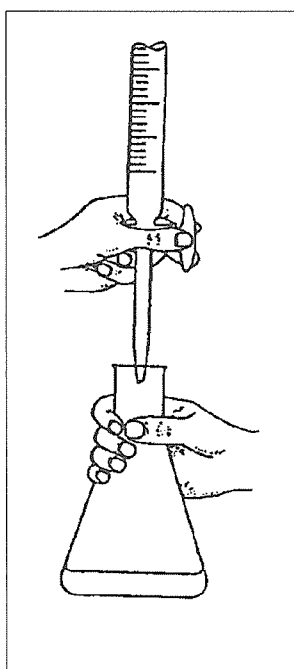
3 Carrying out a titration

Chemicals required Pure water, reagents and indicators as appropriate

Equipment required Burette, burette/retort stand, conical flask, small funnel, white tile, 3 beakers, 25 cm³ pipette, pipette safety filler, conical flask, wash bottle

- Preparing the burette**
- 1 Set up the burette and add a small funnel. Place a white tile on the base of the burette or retort stand.
 - 2 Place the dilute acid in a labelled spare beaker. Add a little of the acid to the burette via the funnel (beware of the acid solution bubbling back if the funnel makes a tight seal with the top rim of the burette).
 - 3 Remove the burette from the stand, rinse the solution around the burette, and finally allow it to pour through the tap end into a beaker reserved for waste.
 - 4 Replace the burette in its stand. Replace the funnel. Place the acid solution in the burette, above the zero mark. Remove the funnel. Adjust the liquid level so that it can be read using the burette scale with any excess liquid going into the waste beaker.

- The titration**
- 5 Place the dilute alkali in another labelled spare beaker.
 - 6 Use a 25 cm³ pipette fitted with a pipette safety filler.
 - 7 Use the pipette filler to withdraw about 25 cm³ of the dilute alkali and expel this into the waste beaker. This rinses the pipette with the dilute alkali.
 - 8 Use the pipette filler to withdraw over 25 cm³ of the alkali and adjust the level so that the pipette reads 25 cm³.
 - 9 Allow this to run into a clean conical flask (the flask having been rinsed with water, first tap and then pure; it does not have to be dry).
 - 10 Add three drops of methyl orange, or phenolphthalein, solution to the conical flask and swirl it gently. (More drops may be required if the chosen indicator is very dilute.)
 - 11 Add the acid from the burette to the flask with constant swirling using the hand technique as shown in the diagram (for a right-handed person) until the acid changes from one colour to an intermediate colour to a final colour. This is a rough result. Towards the end of the titration use a wash bottle to wash down the sides of the conical flask.
 - 12 Wash out the conical flask with tap water and then with a little pure water.
 - 13 Refill the burette to above the zero mark and then set to zero (although consecutive readings can be done).
 - 14 Using the pipette, measure out 25 cm³ of dilute alkali solution and place it in the conical flask with three drops of the indicator solution as before.
 - 15 Add the acid to the conical flask rapidly within 1 cm³ of the value found above. Swirl the flask very gently. Now add the acid drop by drop, swirling each time, until the solution is at the intermediate colour. Note the reading. This is an accurate result.
 - 16 Repeat the accurate titration procedure. The differences should not be more than 0.1 cm³ apart. Ignore the rough titration and any rogue results and work out the average of at least three concordant titres.



3 Carrying out a titration cont.

Choosing the indicator The choice of indicator depends on the type of acid and base being used and also the approximate concentration:

- **Strong acids** include: sulfuric, hydrochloric, nitric and aminosulfonic (sulfamic) acids
- **Weak acids** include: ethanoic acid and potassium hydrogen phthalate
- **Strong bases** include: sodium and potassium hydroxide
- **Weak bases** include: ammonia and sodium carbonate

A laboratory manual could be used to find a suitable indicator. Some recommendations are:

- **strong acid / strong base:**

0.1 mol dm⁻³: (screened) methyl orange or phenolphthalein

0.01 mol dm⁻³: methyl red, bromothymol blue or phenol red

- **weak acid / strong base:** phenolphthalein or thymol blue

- **weak base / strong acid:** (screened) methyl orange or methyl red, bromothymol blue

- **weak acid / weak base:** not recommended

Working out the results You may know how to do this using chemical equations. If in doubt always consult an experienced technician or experienced teacher of chemistry. If still in doubt, CLEAPSS or SSERC can be contacted if your school or college subscribes to their service (see page 5).

Examples 1 and 2 Using 0.1 mol dm⁻³ sodium carbonate as the standard (using methyl orange or other indicator which changes in the same pH range*) The acid is in the burette and the alkali is in the conical flask. A pipette was used to deliver the 0.1 mol dm⁻³ sodium carbonate solution.

$$\text{concentration of hydrochloric acid} = \frac{2 \times 0.1 \times \text{volume of sodium carbonate in the pipette}}{\text{average volume of hydrochloric acid}}$$

$$\text{concentration of sulfuric acid} = \frac{0.1 \times \text{volume of sodium carbonate in the pipette}}{\text{average volume of sulfuric acid}}$$

Example 3 Using 0.1 mol dm⁻³ potassium hydrogen phthalate as the standard

$$\text{concentration of sodium hydroxide} = \frac{0.1 \times \text{volume of sodium hydroxide in the pipette}}{\text{average volume of potassium hydrogen phthalate}}$$

*Methyl orange changes colour when 2 moles of hydrochloric acid react with 1 mole of carbonate. However, if phenolphthalein is used as the indicator, the colour changes when only 1 mole of hydrochloric acid has been used.



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