ADJUSTING pH

Depending on where you live, the natural water supply may be slightly acidic or slightly alkaline. Acidic or alkaline water can corrode pipes, valves and other fittings, resulting in metal ions dissolved in it. To prevent this happening, the pH of water is adjusted in water treatment plants by adding lime (calcium hydroxide) and, sometimes, carbon dioxide. Adding lime increases the pH while adding carbon dioxide decreases the pH.

WHAT YOU HAVE TO DO

You will investigate the effect of adding

- calcium hydroxide, and
- carbon dioxide

to distilled water.

First, you will make the 'samples' and measure their pH. You will then estimate their hardness by titration, using soap solution as the indicator. You'll also investigate the effect of boiling on the water hardness.

EQUIPMENT

water samples

- measuring cylinder
- 4 x test tubes
- test tube racks
- distilled water
- limewater (take care as this is alkaline)
- access to KIPPS apparatus

analysis

- water sample for analysis
- 250 cm^3 stoppered conical flask
- 50 cm^3 burette with stand, clamp and white tile
- 50 cm^3 measuring cylinder
- 250 cm^3 beaker
- glass funnel
- standard soap solution
- pH meter

SAFETY NOTES

Wear protective clothing and eye protection. The standard soap solution contains ethanol and is therefore flammable.

THE THEORY

Water hardness can be estimated by measuring the volume of standard soap solution required to give a lather or froth. Small amounts of calcium hydrogencarbonate, calcium sulfate, magnesium hydrogencarbonate and magnesium sulfate make water 'hard'. More precisely, it is the calcium ions and magnesium ions that cause hardness.

METHOD: SAMPLES

1. Your teacher will tell you which solutions you will test.

2. Label two test tubes:
   - distilled water
   - distilled water + CO_2

   Label two more test tubes with one of the following pairs:
   - limewater
   - limewater + CO_2

   or
   - 50%
   - 50% + CO_2

   or
   - 20%
   - 20% + CO_2

3. Using the measuring cylinder, prepare two samples:
   - 50 cm^3 distilled water

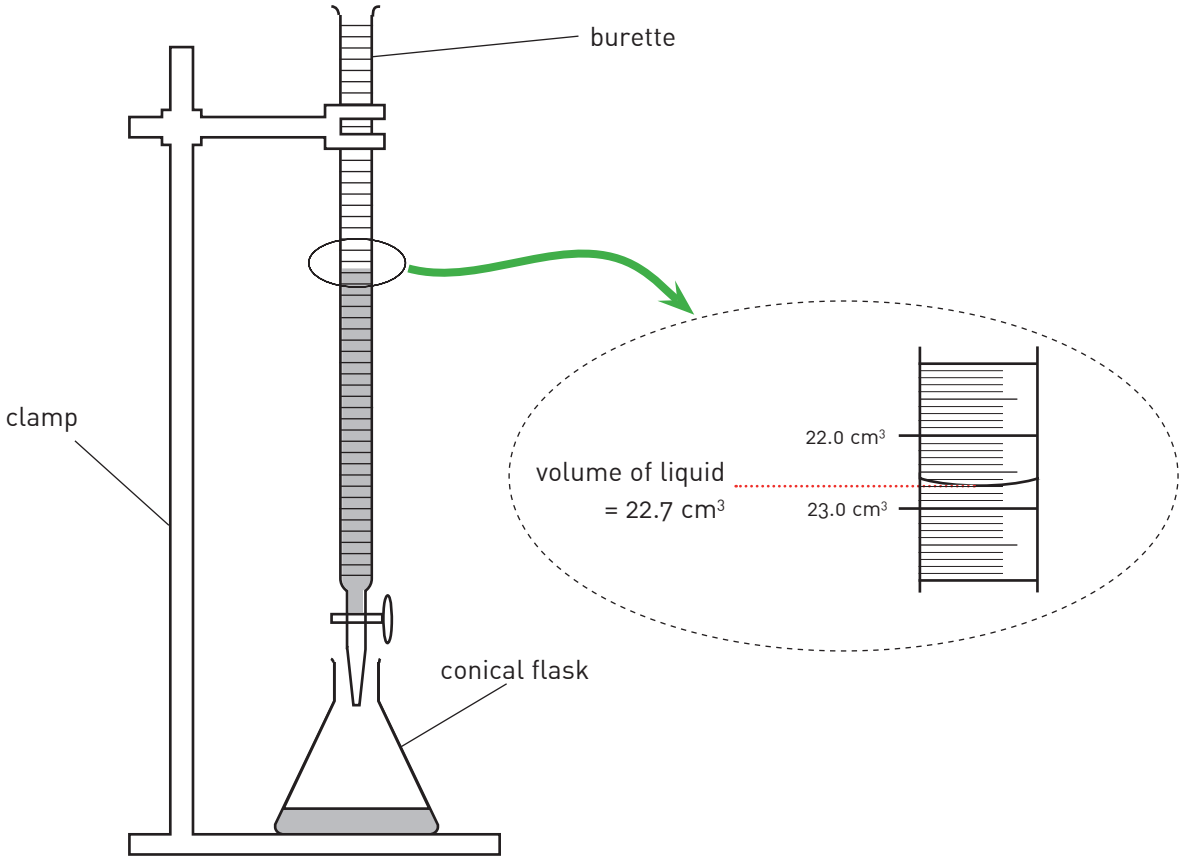
   Prepare two more samples of one of these solutions:
   - 50 cm^3 limewater
   - 25 cm^3 limewater + 25 cm^3 distilled water
   - 10 cm^3 limewater + 40 cm^3 distilled water

   Note: measure out the distilled water into each test tube first.

4. Take the second sample of each dilution (marked + CO2) and bubble carbon dioxide through it for 2-3 minutes (until it goes clear), using the KIPPs apparatus.

METHOD: ANALYSIS

1. Measure the pH of your water sample.
2. Put 50 cm^3 of the water being tested into a 250 cm^3 conical flask.
3. Rinse the burette with distilled water. If there are any drops of water left, the burette may be dirty.
4. Fill the clean burette with the soap solution until just above the 0.00 cm^3 graduation mark. Make sure the burette is vertical before you start to fill it. Use the funnel to help you fill the burette and remove it afterwards.
5. Allow the soap solution to run out until the bottom of the meniscus is just level with the 0.00 cm^3 graduation mark. If the liquid stream comes out sideways the tap might be damaged or blocked. If it is, show your teacher so they can help you unblock it. Make sure there's no air bubble below the tap.



6. Now run the soap solution into the conical flask 1.0 cm^3 at a time. Shake the flask vigorously after each addition. (Put in a bung before shaking.)
7. When the froth takes longer to disappear, add the soap solution 0.5 cm^3 at a time.
8. Continue adding the soap solution until a permanent froth forms.
9. Read the burette and record the volume of soap solution that was added. Make sure your eye is level with the solution and that you take the reading at the bottom of the meniscus (you can hold a piece of white card at an angle behind the burette to help see the meniscus better).
10. Rinse your burette with distilled water.
11. Repeat the procedure for the other samples.

RESULTS

sample	pH	volume of soap solution to make permanent froth / cm^3	volume of soap solution to make permanent froth – distilled water / cm^3	(a) – (b)
		(a)	(b)	
			(b)	
			(b)	

Calculations

The volume of soap solution needed to make a permanent froth is a measure of the hardness. The more soap solution required to make a permanent froth, the harder the water.

Calculate the volume of soap solution needed to react with the salts causing the hardness using these formulae:

volume of soap solution needed to react with salts causing total hardness in 50 cm^3 of water = $a - b$

1 cm^3 standard soap solution is equivalent to 1 mg $CaCO_3$

Total hardness = 20 x ($a - b$) mg $CaCO_3$ per dm^3

Report

Your report should include:

- your results tables
- values for total hardness expressed as mg $CaCO_3$ per dm^3
- values for pH

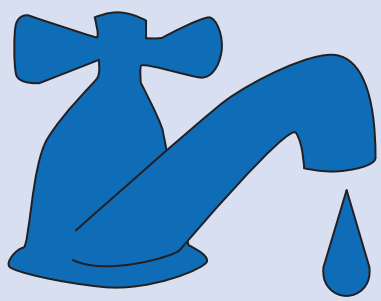
for each water sample.

Write conclusions about the effects of adding:

- calcium hydroxide
- carbon dioxide.

QUESTIONS

1. Why do you use soap solution and not a detergent solution as the 'indictor'? Hint: find out why detergents were developed.
2. How are soaps and detergents made?
3. Find out how the hardness might be determined with greater accuracy.
4. Find out about the two types of hardness: permanent and temporary.
5. Some medical evidence suggests 'some degree of water hardness' is advantageous in drinking water. Find out what these advantages might be (check data from more than one source). http://news.bbc.co.uk/1/hi/health/3396141.stm



HEALTH AND SAFETY

A risk assessment must be made before starting any practical work. Students should wear eye protection. The Wanklyn solution is highly flammable, so avoid naked flames.

NOTES

A range of water samples should be analysed. These may be prepared by the students or in advance. Students will need a sample of at least 50 cm^3 to analyse.

This is not an 'accurate' titration (the end-point is determined only to the nearest 1 cm^3). However, it provides good practice of some essential skills used in volumetric analysis.

The hardness of water depends on its source. Ranges sometimes quoted are:

- Soft 0-75 mg $CaCO_3$ per dm^3
- Moderately hard 75-150 mg $CaCO_3$ per dm^3
- Hard 150-300 mg $CaCO_3$ per dm^3
- Very hard >300 mg $CaCO_3$ per dm^3

In view of the number of solutions to be prepared and analysed, students should work in pairs. Each pair should analyse one solution (plus the CO_2 solution) and the distilled water (plus distilled water + CO_2). Results can be shared. Their results and conclusions should be written up individually.

Time required

This activity will take one session to complete with additional time for writing up and evaluation.

THE THEORY

Total water hardness (the sum of temporary and permanent hardnesses) can be estimated by measuring the volume of standard soap solution required to give a permanent lather or froth. This method can also be used to compare unboiled water (*total* hardness) and boiled water (*permanent* hardness).The difference is a measure of the water's *temporary* hardness.

Soap is a sodium or potassium salt of a fatty acid (a large, naturally-occurring carboxylic acid such as stearic acid or palmitic acid). The sodium and potassium salts are soluble in water. The calcium and magnesium salts of these acids are not. When, for example, sodium stearate is added to a solution containing calcium ions the following reaction occurs:

$2RCOO^-(aq) + Ca_2^+(aq) \rightarrow (RCOO)2Ca(s)$

To estimate hardness, an aqueous solution of soap is reacted quantitatively with calcium and magnesium ions in water. A scum forms (the insoluble calcium and magnesium salts). When all the calcium and magnesium ions have been precipitated, a slight excess of soap produces a permanent lather.

Hardness is expressed as:

milligrams of calcium carbonate per litre of water (mg $CaCO_3$ per dm^3).

TECHNICIAN NOTES

The method requires that 1 cm^3 soap solution ≡ 1 mg $CaCO_3$, necessitating a standard solution, e.g. Wanklyn (Griffin /Fisher Scientific code J/7500/17; Philip Harris B6A71043).This is a solution of potassium oleate in a water/ethanol mixture [HIGHLY FLAMMABLE], and is relatively expensive.

Samples:

- Lime water contains about 0.16 g calcium hydroxide in 100 cm^3 of water. The resulting solution after bubbling CO_2 through has a temporary hardness of 216 mg $CaCO_3$ per dm^3.
- The solutions can be mixed in different proportions to give water samples containing differing amounts of hardness.

TECHNICIAN EQUIPMENT LIST

per student

- eye protection

per group

water samples

- measuring cylinder
- 4 x test tubes
- test tube racks
- distilled water
- limewater
- KIPPS apparatus or other means of generating carbon dioxide

analysis

- water sample for analysis
- 250 cm^3 stoppered conical flask
- 50 cm^3 burette with stand, clamp and white tile
- 50 cm^3 measuring cylinder
- 250 cm^3 beaker
- glass funnel
- Wanklyn standard soap solution [HIGHLY FLAMMABLE]
- pH meter