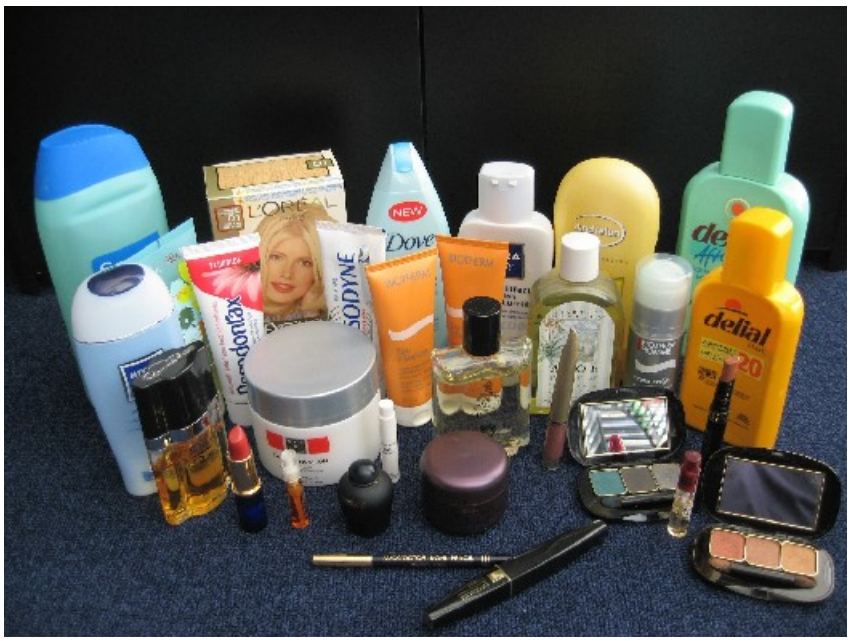
BEAUTI FUL PEOPLE
Analysing cosmetic ingredients



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Summary
We give a lot of attention to our appearance. Cosmetics, jewellery, clothes and tattoos are frequently used to make us look better. A large range of products count as cosmetics, like bath and shower products, deodorants, make-up, shaving products, hair-care products and soap. When you buy a cosmetic product you assume it is safe, especially with cosmetics applied directly on the skin. Authorities check that producers and traders keep to the legal requirements and liabilities laid down in their country. The aim of this project is to investigate a few ingredients in cosmetics, using different techniques, and to determine if the cosmetics meet legal requirements.

Activity type H
Use of scientific knowledge and understanding to solve problems
Working in teams to solve problems
Communication
Resource/ budget management
Time and workload management

Techniques
Potentiometry
Iodometry
Acid-base titration

Precipitation titration
Paper chromatography
TLC
GLC
Gravimetric analysis

Field
Cosmetics

Time
Practical lessons: 720 minutes
Theory lessons: 180 minutes
Out of class time: 180 minutes
Due to the long waiting times, about a week is needed to accomplish this activity.

StandardBase procedures
None

StandardBase techniques
Precipitation titration
Acid/base titration
Potentiometry
Gas Liquid Chromatography
Thin Layer Chromatography
Gravimetric techniques

Other resources
NCV Cosmetica, Zeist, the Netherlands; http://www.ncv-cosmetica.nl
http://ec.europa.eu/enterprise/cosmetics/html/cosm_inci_index.htm
http://www.cir-safety.org/
http://www.fda.gov/fdac/reprints/puffery.html
http://www.cfsan.fda.gov/~dms/cos-chem.html
http://www.cfsan.fda.gov/~dms/cos-prd.html
http://www.hc-sc.gc.ca/cps-spc/person/cosmet/hotlist-liste_e.html
http://www.chemistry.org/portal/a/c/s/1/feature_ent.html?id=17d5f7b2644a11d6e8056ed9fe800100
http://www.ncv-cosmetica.nl/cosmetica/haarkleuringtabel.pdf
http://www.chemsoc.org/chembytes/ezine/1998/houlton.htm

Student's document

BEAUTIFUL PEOPLE: Analysing cosmetic ingredients

ABOUT COSMETICS...

Cosmetics is a general term applied to all preparations used externally to condition and beautify the body, by cleaning, colouring, softening or protecting the skin, hair, nails, lips or eyes. Perfumery is usually excluded from the field of cosmetics, although perfumes are commonly manufactured in coordination with cosmetics.

The use of cosmetics is worldwide and dates from the remotest antiquity. Although it is generally believed that cosmetics as they are now known originated in the Far East, the study of non-industrial cultures indicates the use of cosmetics in every part of the world. The war paint of Native Americans, the tattooing and scarification (making of superficial incisions of the skin) practiced by many peoples (the Maori of New Zealand and numerous African cultures, for instance), and the use of woad (a plant dye used by ancient Britons to paint their bodies blue) are all forms of cosmetic used for psychological intimidation of the enemy as well as adornment.

The earliest known cosmetics come from the First Dynasty of Egypt (about 3100 - 2907 BC). Tombs of this era have yielded unguent jars and, from remains of later periods, it is evident that the unguents were scented. Such preparations, as well as perfumed oils, were extensively used by both men and women to keep the skin supple and unwrinkled in the dry heat of Egypt. Egyptian women also developed the art of decorating the eyes by applying dark green colour to the lower lid and by blackening the lashes and the upper lid with kohl, a preparation made from antimony or soot. It is likely that the Jews adopted the use of cosmetics from the Egyptians, since references to face painting appear in the Old Testament.

By the middle of the first century AD, cosmetics were widely used by the Romans, who employed kohl for darkening eyelashes and eyelids, chalk for whitening the complexion, rouge and depilatories (hair-removing preparations), and pumice for cleaning the teeth. In the Middle Ages, the Crusaders found cosmetics widely used in the Middle East, and it was they who spread the use of cosmetics throughout Europe.

The almost universal use of cosmetics in modern times has grown with the scientific study of the ingredients employed. This research was begun by the French in the 19th century, and led to the development of more and better cosmetics at low cost.

A large variety of cosmetics is generally available today. Cold cream is an emulsion of various oils and waxes and water; it is employed to cleanse and soften the skin. Multi-purpose moisturisers and cleansers are also available. Face powder and dusting powder, based on talcum (powdered magnesium silicate) and zinc oxide, are used to dry and give the skin a satin-like texture. Lip colour, either applied directly as a lipstick or brushed onto the lips, contains cocoa butter or lanolin, and is manufactured in an endless variety of shades, as are rouges (blushers), mixtures of red pigments and starch or finely powdered clay. Bath salts and other bath preparations combine water-softening agents such as sodium carbonate or borax with perfume; bath oils are also a popular skin-softening and perfuming aid. Nail polishes are lacquers or plastics available in many colours. Hair lotions and sprays are used to condition the hair, keep it in place, or make it glossy. Shampoos are based on soap or synthetic detergents.

Hair-colouring dyes, tints, and rinses, available in many shades and colours, are widely used cosmetic products. Henna is a vegetable dye, used for centuries to impart a red tint to the hair. Weak solutions of hydrogen peroxide are often employed as hair bleaches. For colouring the eyebrows and eyelashes, mascara is generally used. This is a compound of gum and black, brown, green, or blue pigment. Sulfides of calcium and barium, which remove hair from the skin, are generally the active agents in cosmetic depilatories. Bronzes are creams that impart a colour to the skin similar to that of suntan.
Cosmetics and perfumery are by no means confined to use by women, as might be assumed. Grooming aids frequently used by men include powders, colognes, and lotions, particularly alcohol-based aftershave lotions; hair tonics, often with an alcohol or quinine base and deodorants.
Annual retail sales of men's and women's toiletries in the Western world today make cosmetics a large and highly profitable industry.
http://www.fashion.vavpycom.net/cosmetichistory.htm

Your brief
Your team (3-5 students) wants to become specialised in analysing cosmetics. When you investigate ingredients in cosmetics, you have to know the requirements and liabilities. Use the table below for the specification of some cosmetics.

Ingredient	**Product**	**Percentage**
Sodium and potassium hydroxides	Skin cleansing products, soap	-
Zinc	Balm	Max 0.1%
Hydrogen peroxide	Tooth whiteners Hair colouring products	- Max 6%
Ammonia	Hair colouring products	-
Sulfites and hydrogen sulfites	Hair decurling products Other hair products	Max 6.7% as SO_2 Max 0.67% as SO_2
Chlorates of the alkali metals	Toothpaste Other products	Max 5% Max 3%
Chloride	Anti-perspire deodorants	Max 3.5%
Ethanol	Perfumes, cleansing tonics, after shaves	-

The techniques you will use are: TLC, GC, paper chromatography, gravimetry and volumetric analyses.

Your brief is to:
- decide which cosmetics are analysed and which experiments you perform, taking into account the techniques you are allowed or requested to use and the size of your team (consult your teacher)
- analyse the chosen cosmetics
- plan time and workload management of this investigation
- calculate the cost of the determinations, bearing in mind the time needed, chemicals used and depreciation of equipment
- write a report with your results, including your plan and the budget
- write a report for consumers

Your investigation

Task 1

Each team member investigates the information on the labels (e.g. ingredients, application and manufacturer) of at least ten products, in local shops. (Go to a shop to gather the information. Do not buy the products yet, you do not have to analyse them all!)

Task 2

List all products, ingredients, analyses and techniques in a table.

Task 3

Write a proposal for your teacher and make sure you can give good reasons why you have chosen specific products and techniques.

Task 4

Consult your teacher and decide which cosmetics you will analyse.

Task 5

Standardisation of titrants: look up an applicable procedure in your textbook. Use the same end point detection for the standardisation as in the determination (visual, potentiometric).

Task 5

Gather the right cosmetic samples, containing the ingredients you want to determine. Each analysis should use a control sample with known content and about the same composition as the real sample (matrix).

Make a plan for all tasks, with a planning grid to show who does what and when. Devise a budget for the experiments (see **Your findings**).

Task 6

Use one or more of the following **student sheets***:*

Determination of ammonia

Determination of chloride

Identification of oxidising agents
Determination of hydrogen peroxide in hair-care products

Identification of chlorates of the alkali metals
Determination of chlorates of the alkali metals

Identification of hydrogen sulfites
Determination of hydrogen sulfites

Identification of free sodium and potassium hydroxides
Determination of free sodium and potassium hydroxides

Determination of methanol in relation to ethanol or propane-2-ol

Determination of zinc

Discuss the plan (time and workload) with your teacher, including the instruments and chemicals required.

Gather information about the techniques you will use.

Remember that a risk assessment must be carried out before you start to work.

Your findings
Gather the results of the experiments. Compare your results within your group and, if possible, with the results of another group (or more groups). Make a comparison table (suggested headings for this table: measurement 1, measurement 2, etc., mean value, standard deviation, value given by manufacturer and the results for the control samples).

Write a report with your results and comparison, and try to answer the next questions:
Do your results match the expected percentages (value given by the manufacturer, value of the control samples)?
Are the results in accordance with your national *Food and Consumer Product* laws? Every country will have its own laws, so look on the Internet or contact your national *Food and Consumer Product* organisation.

Calculate the cost of the analyses.
Example of a report form for one analysis:

Determination of ammonia					
Chemicals	**Quantity/ Amount**	**Price**	**Time needed**	**Instrument costs**	**Energy costs**

Was your estimated budget sufficient? Did you exceed your budget, or was there a deficit on the budget? Explain the differences.

Example of a budget table:

Experiment	**Estimated costs**	**Actual costs**	**Difference**	**Explanation (if possible)**
Ammonia				
Chloride				
Zinc				
etc.				

Was your time schedule correct? Did every member of the group do their share of the work? What problems occurred? How did you solve them?

Example of a time schedule table:

	Management time		**Analysis time**		**etc.**	
Experiment	**Estimate**	**Actual**	**Estimate**	**Actual**		
Ammonia						
Chloride						
Zinc						
etc.						

Write a general report to explain to consumers what is in the cosmetics they use every day, and if the contents meet the legal requirements of your country.

Student self assessment
Use the file: *Evaluating skills.doc* from the ProBase website.

Student sheet: Determination of ammonia

Scope

The aim of this analytical procedure is to determine the amount of ammonia in cosmetic products. It may be used on various hair colouring products.

Principle

Barium chloride solution is added to a test portion of the cosmetic product diluted in an aqueous methanol medium. Any precipitate which may form is filtered or centrifuged off. This procedure avoids the loss of ammonia, during steam distillation, from certain ammonium salts such as the carbonate and hydrogen carbonate and those of the fatty acids, with the exception of ammonium acetate. The ammonia is steam distilled from the filtrate or supernatant and is determined by titration, the end point can be determined by potentiometric method or with a colour acid/base indicator.

Reactions:

$$NH_3 + H_3O^+ \rightarrow NH_4^+ + H_2O$$

$$NH_4^+ + OH^- \rightarrow NH_3 + H_2O$$

Apparatus

Equipment - Instruments

- centrifuge with stoppered 100 mL bottles
- steam distillation apparatus
- pH-millivolt meter with glass and reference (Ag/AgCl/Cl^-) electrode

Equipment - Glassware and other equipment

- usual laboratory glassware and equipment

Materials

- methanol
- barium chloride dihydrate
- boric acid
- ethanol
- sulfuric acid
- sodium hydroxide
- methyl red
- methylene blue

Reagent solutions

- Barium chloride dihydrate, 250 g/L solution: Dissolve 250 g of solid barium chloride dehydrate in distilled water and fill up to 1 L
- Boric acid 40 g/L solution: Dissolve 40 g of solid boric acid in distilled water and fill up to 1 L
- Sulfuric acid, 0.25 mol/L solution: Dilute 13.9 mL of concentrated sulfuric acid in distilled water and fill up to 1 L
- Sodium hydroxide, 0.5 mol/L solution: Dissolve 20 g of solid NaOH in distilled water and fill up to 1 L
- Indicator, if required: Mix 5 ml of 1 mg/L methyl red solution in ethanol with 2 mL of 0.1 mg/L methylene blue solution in water

Procedure

1. Weigh into a 100 mL standard flask a mass (m) of the sample corresponding to maximum 150 mg of ammonia. Add 10 mL of water, 10 mL of methanol and 10 mL of barium chloride solution. Fill up to 100 mL with methanol. Mix and leave overnight in a refrigerator (5 °C).

2. Filter, or centrifuge, the still cold solution in closed tubes for 10 minutes, so as to obtain a clear filtrate or supernatant layer.

3. Pipette 40 mL of this clear solution into the steam distillation apparatus, followed by 0.5 mL of antifoam liquid, where appropriate.

4. Distil and collect 200 mL of distillate in a 300 mL conical flask containing 10 mL of standard sulfuric acid and 0.1 mL of indicator.

5. Back titrate the excess acid with standard sodium hydroxide solution.

NB: For potentiometric determination, collect 200 mL of distillate in a 250 mL beaker containing 25.00 mL of boric acid solution and titrate with standard sulfuric acid.

Standardisation of 0.5 mol/L sodium hydroxide and 0.25 mol/L sulfuric acid is necessary.

Calculations

Calculation - back titration

$$w_{NH_3} = \frac{[(2 \times V_{H_2SO_4} \times c_{H_2SO_4}) - (V_{NaOH} \times c_{NaOH})] \times 17 \times 2.5}{m} \times 100\%$$

V_{NaOH} the volume (in millilitres) of the sodium hydroxide solution used
c_{NaOH} concentration of sodium hydroxide solution in mol/L
$V_{H_2SO_4}$ the volume (in millilitres) of the sulfuric acid solution used
$c_{H_2SO_4}$ concentration of sulfuric acid solution in mol/L
m the mass (in milligrams) of the sample taken
17 molar mass of ammonia in g/mol
2 mole ratio for sulfuric acid and sodium hydroxide
2.5 dilution factor

Calculation - direct potentiometric titration

$$w_{NH_3} = \frac{V_{H_2SO_4} \times c_{H_2SO_4} \times 17 \times 2.5}{m} x100\%$$

$V_{H_2SO_4}$ the volume (in millilitres) of the sulfuric acid solution used
$c_{H_2SO_4}$ concentration of sulfuric acid solution in mol/L
m the mass (in milligrams) of the sample
17 molar mass of ammonia in g/mol
2.5 dilution factor

Student sheet: Determination of chloride

Scope
The aim of this analytical procedure is to determine the amount of chloride present as chloride ion in aluminium zirconium chloride hydroxide complexes in non-aerosol antiperspirants.

Principle
Chloride ion in the product is determined by potentiometric titration using a standard silver nitrate solution.
Reaction:

$$Cl^- + Ag^+ \rightarrow AgCl(s)$$

Apparatus
Equipment - Instruments
- heater with magnetic stirrer
- pH/millivolt meter suitable for potentiometric titration, with silver electrode and calomel reference electrode

Equipment - Glassware and other equipment
- usual laboratory glassware and equipment

Materials
- nitric acid, concentrated
- acetone
- silver nitrate

Reagent solutions
- Silver nitrate solution: (0.1 mol/ L): Accurately weigh about 4.25 g of solid $AgNO_3$ and dissolve it in 250 mL of distilled water in a volumetric flask. Store the solution in a brown bottle.
- Nitric acid solution: Add 25 mL concentrated nitric acid to 250 mL of water in a beaker, stirring continuously. Transfer this solution to a 500 mL volumetric flask and fill up to volume with water.

Procedure
1. Weigh accurately into a 250 mL beaker approximately 1.0 g (*m*) of a homogenous sample of the product. Add 80 mL of water and 20 mL of the nitric acid solution.

2. Place the beaker on a heater with a magnetic stirrer. Commence stirring and heat to boiling. To prevent rapid drying, place a watchglass on top of the beaker. Boil for five minutes, remove beaker from heat and cool to room temperature.

3. Add 10 mL of acetone and dip the electrodes below the surface of the solution and commence stirring. Titrate potentiometrically with 0.1 mol/L silver nitrate solution and determine the endpoint (*V*).

Standardisation of 0.1 mol/L silver nitrate is necessary.

Calculation
Calculate the chloride content of the sample, in percentage by mass, using the formula:

$$w_{Cl_2} = \frac{35.45 \times V_{AgNO_3} \times c_{AgNO_3}}{m} \times 100\%$$

m	mass in milligrams of the sample taken for analysis
V_{AgNO_3}	volume of 0.1 mol/L silver nitrate, in millilitres, titrated at the endpoint
c_{AgNO_3}	concentration of 0.1 mol/L silver nitrate
35.45	atomic mass of chloride in g/mol

Student sheet: Identification of oxidising agents

Scope
The aim of this identification procedure is to detect oxidising agents (persulfates, bromates etc.) other than hydrogen peroxide present in samples, like hair-care products.

Principle
Sodium persulfate, potassium persulfate and ammonium persulfate, potassium bromate, sodium bromate and hydrogen peroxide − whether or not originating from barium peroxide − are identified by means of descending paper chromatography, use being made of two developing solvents.

Apparatus
Equipment - Instruments
- apparatus for descending paper chromatography

Equipment - Glassware and other equipment
- chromatography paper (Whatman paper No 3 and No 4 or their equivalents)
- standard flasks, 100 mL
- micropipette 1 µL
- fluted filters

Materials
- sodium persulfate
- potassium persulfate
- ammonium persulfate
- potassium bromate
- sodium bromate
- potassium iodide
- ethanol absolute
- methanol
- toluene
- 3-methyl butan-1-ol
- starch
- hydrochloric acid, concentrated
- hydrogen peroxide

Reagent solutions
- 5 g/L aqueous reference solutions of the following compounds: sodium persulfate, potassium persulfate, ammonium persulfate, potassium bromate, sodium bromate: Dissolve 0.5 g of the compounds in distilled water and fill up to 100 mL
- Reference solution hydrogen peroxide: Dilute 1.6 mL hydrogen peroxide solution (300 g/L) in distilled water and fill up to 100 mL
- Developing solvent A: Take 400 mL of ethanol and fill up to 500 mL with distilled water
- Developing solvent B, toluene/methanol/3-methyl butan-1-ol/water: Mix 170 mL toluene, 190 mL methanol, 90 mL 3-methyl butan-1-ol and 50 mL water

- Detecting agent X, 100 g/L aqueous solution of potassium iodide, to be freshly prepared immediately before use: Dissolve 10 g of potassium iodide in distilled water and fill up to 100 mL
- Detecting agent Y: 10 g/L aqueous solution of starch. Dissolve 1 g starch in 100 mL boiling distilled water
- Detecting agent Z: Dissolve 27.7 mL concentrated hydrochloric acid in distilled water and fill up to 100 mL
- 4 mol/L hydrochloric acid: Dilute 333 mL of concentrated hydrochloric acid in distilled water and fill up to 1 L

Preparation

Preparation of sample

Water soluble products

Make two solutions of each sample by dissolving 1 g and 5 g of the product, respectively in 100 mL of water. Use 1 µL of each of these solutions for carrying out the paper chromatography.

Products sparingly soluble in water

Weigh out 1 g and 5 g of the sample and disperse in 50 mL of water, fill up to 100 mL with water in each case and mix. Filter the two dispersions over a fluted filter and use 1 µL of each of the filtrates in order to carry out the paper chromatography.

Prepare once again two dispersions of each sample by dispersing 1 g and 5 g in 50 mL of water, acidify with dilute hydrochloric acid, and fill up with water to 100 mL and mix. Filter the dispersions through a fluted filter and use 1 µL of the two filtrates in order to carry out the paper chromatography.

Creams

Disperse 5 g and 20 g of each product in 100 mL of water and use the dispersions to carry out the paper chromatography.

Procedure

1. Put an appropriate quantity of developing solvents A and B into two separate chromatography tanks in order to carry out the descending paper chromatography. Saturate the chromatography tanks with solvent vapour.

2. Apply 1 µl of one sample solution and 3 (or more if possible) reference solutions prepared to each starting point on a strip of chromatography paper (Whatman No 3 or equivalent) 40 cm long and 20 cm wide or another suitable format and evaporate the solution in air.

3. Place the chromatography strip in the chromatography tank filled with developing solvent A and develop until the solvent front has advanced 35 cm (about 15 hours).

4. Repeat the procedure (use different reference solutions) using chromatography paper (Whatman No 4 or equivalent) and developing solvent B. Chromatograph until the solvent front has advanced 35 cm (about five hours).

5. After development, remove the chromatograms and dry them in air.

6. Reveal the spots in the chromatogram by spraying it successively with:
- detecting agent X followed shortly thereafter by detecting agent Y. The spots of the persulfates will now appear first in the chromatogram and will be followed by the hydrogen peroxide spots. Mark the spots with a pencil.

- detecting agent Z on the chromatograms sprayed by detecting agent A and B. The presence of bromates will now be indicated by greyish blue spots in the chromatogram.

Under the above mentioned conditions pertaining to developing solvents A and B, the R_f values of the reference substances are approximately as follows:

	Developing solvent A	**Developing solvent B**
Sodium persulfate	0.40	0.10
Potassium persulfate	0.40	0.02 + 0.05
Ammonium persulfate	0.50	0.10 + 0.20
Sodium bromate	0.40	0.20
Potassium bromate	0.40	0.10 + 0.20
Hydrogen peroxide	0.80	0.80

Student sheet: Determination of hydrogen peroxide

Scope
The aim of this analytical procedure is to determine the amount of hydrogen peroxide in cosmetics, like hair-care products

Principle
The iodometric determination of hydrogen peroxide is based on the following reaction:

$$H_2O_2 + 2H_3O^+ + 2I^- \rightarrow I_2 + 4H_2O$$

This conversion proceeds slowly but can be accelerated by the addition of ammonium molybdate. The iodine formed is determined titrimetrically with sodium thiosulfate and is a measure of the hydrogen peroxide content.
Titration:

$$I_2 + 2S_2O_3^{2-} \rightarrow 2I^- + S_4O_6^{2-}$$

Apparatus
Equipment - Glassware and other equipment
- burette, 50 mL
- usual laboratory glassware

Materials
- sulfuric acid, concentrated
- potassium iodide
- ammonium molybdate
- sodium thiosulfate
- starch

Reagent solutions
- 2 mol/L sulfuric acid: Dilute 111 mL of concentrated sulfuric acid in distilled water and fill up to 1 L
- Ammonium molybdate solution (20 g/L): Dissolve 2 g ammonium molybdate in distilled water and fill up to 100 mL
- Potassium iodide solution (100 g/L): Dissolve 10 g potassium iodide in distilled water and fill up to 100 mL (it has to be freshly prepared.)
- 0.1 mol/L sodium thiosulfate: Dissolve 12.4 g $Na_2S_2O_3.5H_2O$ in 0.5 L distilled water
- 10 g/L aqueous solution of starch: Dissolve 1 g starch in 100 mL boiling distilled water

Procedure
1. Into a 100 mL beaker weigh out 10 g (m) of the product, containing about 0.6 g of hydrogen peroxide. Transfer the contents with water into a 250 mL standard flask, fill up to the mark with water and mix.

2. Pipette 10.00 mL of the sample solution into a 250 mL conical flask and add successively 100 mL of 2 mol/L sulfuric acid, 20 mL of potassium iodide solution and three drops of ammonium molybdate solution.

3. Titrate the iodine formed immediately with 0.1 mol/L sodium thiosulfate solution and just before the end point is reached add a few millilitres of starch

solution as indicator. Record the consumption of 0.1 mol/L sodium thiosulfate in millilitres (V).

In the same way, carry out a blank determination, replacing the 10 ml of the sample solution by 10 ml of water. Record the consumption of 0.1 mol/L sodium thiosulfate in the blank determination (V_0 mL).

Standardisation of 0.1 mol/L sodium thiosulfate is necessary

Calculation
Calculate the hydrogen peroxide content of the product as a percentage by mass with the aid of the following formula:

$$w_{H_2O_2} = \frac{(V_{Na_2S_2O_3} - V_0) \times c_{Na_2S_2O_3} \times 0.5 \times 34.0 \times 25}{m} \times 100\%$$

m	the mass in milligrams of product analysed
V_0	the consumption in millilitres of 0.1 mol/L thiosulfate solution in the blank determination
$V_{Na_2S_2O_3}$	the consumption in millilitres of 0.1 mol/L thiosulfate solution in the titration of the sample solution
$c_{Na_2S_2O_3}$	concentration of 0.1 mol/L sodium thiosulfate solution
0.5	mole ratio of H_2O_2 and $S_2O_3^{2-}$
25	dilution factor
34.0	molar mass of H_2O_2 in g/mol

Student sheet: Identification of chlorates of the alkali metals

Scope
The aim of this identification procedure is to detect chlorates of alkali metals present in toothpastes and other cosmetic products.

Principle
Chlorates are separated from other halates by thin layer chromatography and identified by the oxidation of iodide to form iodine.

Apparatus
Equipment - Glassware and other equipment
- normal equipment for thin layer chromatography
- ready-for-use cellulose thin-layer plates (0.25 mm)

Materials
- potassium chlorate
- potassium bromate
- potassium iodide
- ammonia, concentrated
- acetone
- butanol
- hydrochloric acid, concentrated
- starch

Reagent solutions
- Reference solutions: aqueous solutions of potassium chlorate, bromate and iodate (2 g/L) prepared freshly: Dissolve 20 mg of potassium chlorate, bromate and iodate in distilled water and fill up to 10 mL
- Development solvent: ammonia/acetone/1-butanol: Mix 60 mL concentrated ammonia, 130 mL acetone and 30 mL 1-butanol
- Potassium iodide, aqueous solution (50 g/L): Dissolve 5 g potassium iodide in distilled water and fill up to 100 mL
- Starch solution (10 to 50 g/L): Dissolve 1-5 g starch in 100 mL boiling distilled water
- 1 mol/L hydrochloric acid: Dilute 83.3 mol of concentrated hydrochloric acid in distilled water and fill up to 1 L.

Procedure
1. Extract about 1 g of the sample with water, filter, and dilute to about 25 mL.

2. Deposit 2 µL on the plate of the solution together with 2 µL aliquots of each of the three reference solutions.

3. Place the plate in a tank and develop by ascending chromatography about three-quarters of the length of the plate with the developing solvent.

4. Remove from the tank and allow the solvent to evaporate. (NB: This may take up to two hours.)

5. Spray the plate with potassium iodide and allow drying for about five minutes.

6. Spray the plate with starch solution and allow drying for about five minutes.

7. Spray the plate with hydrochloric acid.

If the chlorate is present a blue spot (possibly a brown spot) will appear after half an hour with R_f value approximately 0.7 to 0.8.

Halates	$\boldsymbol{R_f}$
Iodate	0 to 0.2
Bromate	0.5 to 0.6
Chlorate	0.7 to 0.8

It should be noted that bromates and iodates give immediate reaction. Care should be taken not to confuse spots from bromates and chlorates.

Student sheet: Determination of chlorates of the alkali metals

Scope
The aim of this analytical procedure is to determine the amount of chlorates of alkali metals present in toothpastes and other cosmetic products.

Principle
Chlorate is reduced by zinc powder under acid conditions. The chloride formed is measured by potentiometric titration using a silver nitrate solution. A similar determination before reduction permits the possible presence of halides.
Reduction:

$$6H_3O^+ + ClO_3^- + 3Zn \rightarrow Cl^- + 3Zn^{2+} + 9H_2O$$

Titration:

$$Cl^- + Ag^+ \rightarrow AgCl(s)$$

Apparatus
Equipment - Instruments
- centrifuge
- pH/millivolt meter suitable for potentiometric titration
- silver electrode and calomel reference electrode

Equipment - Glassware and other equipment
- normal laboratory equipment

Materials
- zinc powder
- silver nitrate
- acetic acid, concentrated

Reagent solutions
- Acetic acid solution: Dilute 800 g glacial acetic acid to 1 L with distilled water
- Silver nitrate solution (0.1 mol/ L): Accurately weigh about 4.25 g of solid $AgNO_3$ and dissolve it in 250 mL of distilled water in a volumetric flask. Store the solution in a brown bottle.

Preparation
Preparation of sample
Weigh accurately a mass of approximately 2 g (m) in centrifuge tube. Add about 15 mL acetic acid solution and mix carefully. Wait 30 minutes and centrifuge for 15 minutes at 2000 rpm. Transfer the supernatant solution to a 50 mL volumetric flask. Repeat centrifuging twice by adding 15 mL acetic acid solution to the residue.
Collect the solution containing chlorate in the same volumetric flask. Fill to the mark with acetic acid solution (*solution A*).

Reduction of chlorate
Take 20 mL of solution from the 50 mL volumetric flask (solution A) and add 0.6 g of zinc powder. Bring to the boil in a flask fitted with a condenser tube. After 30 minutes boiling, cool and filter. Rinse the flask with water. Filter and combine the filtrate with the rinses (*solution B*).

Procedure

1. Titrate 20 mL of solution B with silver nitrate by using the pH/millivolt meter.

2. Titrate in the same way 20 mL of solution A with silver nitrate.

NB: If the product contains bromine or iodine derivatives which can release bromides or iodides after reduction, the titration curve will have several inflexion points. In this case the volume of the titrated solution corresponding to chloride is the difference between the last and the penultimate inflection points.

Standardisation of 0.1 mol/L silver nitrate is necessary.

Calculation

The content of chlorate of the sample w is calculated by the formula:

$$w_{(\text{chlorate})} = \frac{(V_B - V_A) \times c_{AgNO_3} \times 83.5 \times 25}{m} \times 100\%$$

V_B volume in mL of silver nitrate solution used to titrate 20 mL solution B
V_A volume, in mL of silver nitrate solution used to titrate 20 mL of solution A
c_{AgNO_3} concentration of silver nitrate standard solution in mol/L
m mass of sample, in milligrams
83.5 molar mass of ClO_3^- in g/mol
2.5 dilution factor

Student sheet: Identification of hydrogen sulfites

Scope
The aim of this identification procedure is to detect inorganic and hydrogen sulfites present in toothpastes and other cosmetic products.

Principle
The sample is heated in hydrochloric acid, and the sulfur dioxide liberated is identified by its effect on an indicator paper.

Apparatus
Equipment - Glassware and other equipment
- flask (25 mL) fitted with a short reflux condenser
- normal laboratory equipment

Materials
- hydrochloric acid
- potassium iodate starch paper

Reagent solutions
- 4 mol/L hydrochloric acid: Dilute 333 mL of concentrated hydrochloric acid in distilled water and fill up to 1 L
- Potassium iodate starch paper or other suitable alternative

Procedure
1. Place about 2.5 g of sample in the 25 mL flask with 10 mL of 4 mol/L hydrochloric acid. Mix and heat to boiling.

2. Test for the emission of sulfur dioxide by indicator paper.

Student sheet: Determination of hydrogen sulfites

Scope
The aim of this analytical procedure is to determine the amount of inorganic and hydrogen sulfites present in cosmetic products.

Principle
After acidification of the sample, the sulfur dioxide liberated is distilled into a solution of hydrogen peroxide. The sulfuric acid formed is titrated against a standardised sodium hydroxide solution.
Reactions:

$$H_2SO_3 \rightarrow H_2O + SO_2$$

$$H_2O_2 + SO_2 + 2H_2O \rightarrow 2H_3O^+ + SO_4^{2-}$$

Titration:

$$H_3O^+ + OH^- \rightarrow 2H_2O$$

Apparatus
Equipment - Instruments
- distillation apparatus

Equipment - Glassware and other equipment
- normal laboratory equipment

Materials
- potassium iodate
- hydrogen peroxide
- sodium hydroxide
- phosphoric acid, concentrated
- methanol
- ethanol
- methyl red
- methylene blue
- nitrogen

Reagent solutions
- Hydrogen peroxide 2 g/L (prepare daily): Dissolve 0.67 mL hydrogen peroxide (300 g/L) in distilled water and fill up to 100 mL
- Sodium hydroxide (0.01 mol/L) solution: Dissolve 0.4 g of solid NaOH in distilled water and fill up to 1 L
- Indicator: Mix equal volumes of methyl red (0.3 g/L in ethanol) and methylene blue (0.5 g/L in ethanol); filter the solution

Procedure
1. Weigh accurately about 2.5 g of sample into the distillation flask. Add 60 mL of water and 50 mL of methanol and mix.

2. Place 10 mL of hydrogen peroxide solution (2 g/L), 60 mL of water and a few drops of methyl red/methylene blue indicator in the distillation receiver. Add a few drops of sodium hydroxide 0.01 mol/L until the indicator turns green.

3. Assemble the apparatus and adjust the nitrogen flow to about 60 bubbles per minute. Run 15 mL of concentrated phosphoric acid from the funnel into the

distillation flask. Heat rapidly to boiling and then simmer gently for a total time of 30 minutes.

4. Detach the distillation receiver. Rinse the tube and then titrate with sodium hydroxide solution until the indicator turns green.

Standardisation of 0.01 mol/L sodium hydroxide is necessary.

Calculation

Calculate the content of sulfite or hydrogen sulfite as sulfur dioxide by mass in the sample by the expression:

$$w_{SO_2} = \frac{V_{NaOH} \times c_{NaOH} \times 64 \times 0.5}{m} \times 100\%$$

c_{NaOH}	concentration of sodium hydroxide solution in mol/L
V_{NaOH}	volume of sodium hydroxide required for titration in mL
m	mass of sample in milligrams.
64	molar mass of sulfur dioxide in g/mol
0.5	mole ratio of H_2SO_3 and NaOH

Student sheet:
Identification of free sodium and potassium hydroxides

Scope
The aim of this identification procedure is to detect significant amounts of free sodium and/or potassium hydroxides present in cosmetic products.

Principle
The pH of the solution or dispersion of the sample is measured.

Apparatus
Equipment - Instruments
- pH/millivolt meter suitable for potentiometric titration
- glass membrane electrode and calomel reference electrode

Equipment - Glassware and other equipment
- usual laboratory glassware

Materials
- disodium tetra borate decahydrate

Reagent solutions
- Standard alkaline buffer solution pH 9.18 at 25 °C, 0.05 mol/l disodium tetra borate decahydrate: Dissolve 4.7658 g disodium tetra borate decahydrate in distilled water, fill up to the mark in a 250 mL standard flask and mix.

Procedure
Calibrate the pH meter with the electrodes using the standard buffer solution pH 9.18. Prepare a 10 % solution or dispersion of the product to be analysed, in water, and filter. Measure the pH.

If the pH is 12 or over, a quantitative determination must be carried out.

Student sheet:
Determination of free sodium and potassium hydroxides

Scope
The aim of this analytical procedure is to determine free sodium and/or potassium hydroxides in cosmetic products like hair straightener preparations and nail cuticle solvent preparations.

Principle
The free sodium or potassium hydroxide is determined with potentiometric titration with an acid. Ammonia is removed in the method by evaporation at reduced pressure at room temperature.
Where salts of weak acids are present, only the first part of the curve, to the first of these points of inflection, corresponds to the neutralisation of hydroxyl ion coming from free sodium or potassium hydroxide.
An alternative procedure for titration in alcohol is given where excessive interference from salts of weak inorganic acids is indicated.

Apparatus
Equipment - Instruments
- pH/millivolt meter suitable for potentiometric titration, preferably with recorder
- glass electrode and calomel reference electrode.

Equipment - Glassware and other equipment
- usual laboratory glassware

Titration in aqueous medium
Materials
- hydrochloric acid, concentrated

Reagent solutions
- mol/L hydrochloric acid: Dilute 8.3 mL of concentrated hydrochloric acid in distilled water and fill up to the mark in a 1 L standard flask and mix 1 L

Procedure
1. Weigh accurately into a 150 mL beaker a test portion of between 0.5 and 1.0 g. If ammonia is present add a few anti-bumping granules.

2. Place the beaker in a vacuum desiccator and evacuate using a water pump until the odour of ammonia is no longer detectable (about three hours).

3. Add 100 mL water, dissolve or disperse the residue and titrate with the 0.1 mol/L hydrochloric acid solution, recording the change in pH.

Standardisation of 0.1 mol/L hydrochloric acid is necessary.

Calculation
Identify the points of inflection on the titration curves. Where the first point of inflection occurs at a pH below 7, the sample is free of sodium or potassium hydroxide.

Where there are two or more points of inflection in the curve, only the first is relevant.

Note the volume of titrant to this first point of inflection.

$$w_{NaOH} = \frac{V_{HCl} \times c_{HCl} \times 40}{m} \times 100\%$$

V_{HCl} volume titrant HCl in mL
c_{HCl} concentration titrant HCl in mol/L
m mass of sample in mg
40.0 molar mass NaOH in g/mol

Titration in propane-2-ol
The situation may arise in which, despite indications of the presence of a significant quantity of sodium and/or potassium hydroxides, the titration curve fails to show a distinct point of inflection. In such a case, the determination should be repeated in propane-2-ol.

Materials
- hydrochloric acid, concentrated

Reagent solutions
- 1 mol/L hydrochloric acid: Dilute 83.3 mL of concentrated hydrochloric acid in distilled water and fill up to 1 L
- 0.1 mol/L hydrochloric acid in propane-2-ol prepared immediately before use by diluting 100 mL 1.0 mol/L aqueous hydrochloric acid with propane-2-ol to 1 L

Procedure
1. Weigh accurately into a 150 mL beaker a test portion of between 0.5 and 1.0 g. If ammonia is present add a few anti-bumping granules.

2. Place the beaker in a vacuum desiccator and evacuate using a water pump until the odour of ammonia is no longer detectable (about three hours).

3. Add 100 mL propane-2-ol, dissolve or disperse the residue and titrate with the 0.1 mol/L hydrochloric acid in propane-2-ol. Record the change in apparent pH.

Standardisation of 0.1 mol/L hydrochloric acid is necessary.

Calculation
See calculation in aqueous medium.

Student sheet:
Determination of methanol in relation to ethanol or propane-2-ol

Scope
The aim of this analytical procedure is to determine methanol in all kinds of (non aerosol) cosmetics

Principle
The determination is carried out by gas chromatography.

Apparatus
Equipment - Instruments
- gas chromatograph: with polar column Tenax, nitrogen carrier gas and FID detector (capillary column GC will give even better separation)

Equipment - Glassware and other equipment
- volumetric flasks, 100 mL
- pipettes, 2 mL, 20 mL, 0 to 1 mL
- micro syringes 0 to 100 µL and 0 to 5 µL

Materials
- methanol
- ethanol, absolute
- propane-2-ol

Preparation
Preparation of sample
Dilute the sample with water to a level of 1 to 2% ethanol or propane-2-ol in a 100 mL volumetric flask.

Preparation of gas chromatograph
Prepare the gas chromatograph as described in the user manual. Make sure that temperatures of oven, injector and detector are stabilised.

Column:	1.6 metre Tenax 80/100 mesh by GC-use
Carrier gas:	Nitrogen 20 mL per minute
Oven:	100 °C
Detector:	FID 150 °C
Injector:	150 °C
Range:	10^{-9}
Attenuation:	1-1024 to give max peak heights

Note: Use the attenuator to give the largest peaks possible. This will give better measurements of the peak heights on paper.

Procedure
1. Use the following standard mixtures for the gas chromatography procedure. Prepare these mixtures by measuring with micro syringe and pipette, but find the exact amount by immediate weighing of the pipette or flask after each addition.

		Methanol / µL	Ethanol or propane-2-ol / mL	Water added to a volume of / mL
1	Approximately 2.5%	50	2	100
2	Approximately 5.0%	100	2	100
3	Approximately 7.5%	150	2	100
4	Approximately 10.0%	200	2	100

2. Inject 2 to 3 µL of the standard mixtures and the samples into the gas chromatograph. Measure the height of all the peaks.

3. Inject 2 to 3 µL of the samples and the samples into the gas chromatograph. Measure the height of all the peaks.

Calculation
Calculate peak area ratio (methanol/ethanol) or (methanol/propane-2-ol) of each mixture.

Make a calibration graph:
x-axis: % methanol
y-axis: peak area ratio (methanol/ethanol) or (methanol/propane-2-ol)

The standard graph must be a straight line.
Read from the graph the methanol percentage for all samples.

Note:
When a percentage of 7.08% for a sample is read on the *x*-axis of the calibration curve, you need to consider the dilution factor of the sample, for example 25 mL sample in 100 mL flask: $f = 100/25 = 4$
The actual amount of methanol in the undiluted sample would be
$7.08 \times f = 7.08 \times 4 = 28.32$ % methanol.

Student sheet: Determination of zinc

Scope
The aim of this analytical procedure is to determine zinc present as chloride, sulfate or 4-hydroxybenzenesulfonate, or as the total of several of these zinc salts, in cosmetics.

Principle
Zinc present in solution is precipitated in an acid medium as zinc(2-methyl-8-quinolinol). After filtration, the precipitate is dried and weighed.
Reaction:

$$Zn^{2+} + 2C_{10}H_8NOH + 2H_2O \rightarrow (C_{10}H_8NO)\text{-}Zn\text{-}(ONH_8C_{10}) + 2H_3O^+$$

Apparatus
Equipment - Instruments
- drying oven regulated to a temperature 150 ± 2 °C
- hotplate

Equipment - Glassware and other equipment
- glass filter crucibles G-4
- vacuum flasks, 500 mL
- water-jet pump
- thermometer graduated from 0 to 100 °C
- desiccator with a suitable desiccant and humidity indicator e.g. silica gel or equivalent
- filter paper, Whatman No 4 or equivalent
- usual laboratory glassware

Materials
- 2-methyl-8-quinolinol
- ammonium acetate
- glacial acetic acid
- ammonia, concentrated

Reagent solutions
- Ammonia solution: Transfer 240 g of concentrated ammonia into a 1000 mL standard flask, fill up to the mark with distilled water and mix
- 0.2 mol/L ammonium acetate solution: Dissolve 15.4 g of ammonium acetate in distilled water, fill up to the mark in a 1000 mL standard flask and mix
- 2-methyl-8-quinolinol solution: Dissolve 5 g of 2-methyl-8-quinolinol in 12 mL of glacial acetic acid and transfer with distilled water into a 100 mL standard flask. Fill up to the mark with distilled water and mix
- pH indicator paper (0-6)

Procedure
1. Weigh out into a 400 mL beaker, 5 to 10 g (m), containing about 50 to 100 mg of zinc, of the sample to be analysed. Add 50 mL of distilled water and mix. Filter, with the aid of a vacuum pump if necessary, and retain the filtrate.

2. Repeat the extraction step with a further 50 mL of distilled water. Filter and combine the filtrates.

3. For every 10 mg of zinc present in the solution, add 2 mL of the 2-methyl-8-quinolinol solution and mix. Dilute the mixture with 150 mL of distilled water.

4. Bring the temperature of the mixture up to 60 °C and add 45 mL of 0.2 mol/L ammonium acetate solution, stirring constantly. Adjust the pH of the solution to 5.5, with the ammonia solution, stirring constantly; use indicator paper to measure the pH of the solution.

5. Allow the solution to stand for 30 minutes. Filter with the aid of a water-jet pump through a G-4 filter crucible which has been dried beforehand (150 °C) and weighed after cooling (m_0), and wash the precipitate with 150 mL of distilled water at 95 °C.

6. Place the crucible in a drying oven regulated to 150 °C and dry for one hour.

7. Remove the crucible from the drying oven, place it in a desiccator and, when it has cooled to room temperature, determine the mass (m_1).

Calculation
Calculate the zinc content of the sample as a percentage by mass with the aid of the following formula:

$$w_{Zn} = \frac{(m_1 - m_0) \times 65.38}{m \times 381.76} \times 100\%$$

m	the mass in grams of the sample taken
m_0	the mass in grams of the empty and dry filter crucible
m_1	the mass in grams of the filter crucible with precipitate
65.38	molar mass zinc in g/mol
381.76	molar mass zinc (2-methyl-8-quinolinol) in g/mol

Teacher's document

Overview
Students work in a small team, working in a cosmetic analysis laboratory. The team has been asked to investigate a few ingredients in cosmetics, using different techniques, and to determine if the cosmetics meet legal requirements.

The team must plan the activity, estimate its cost and write a report to explain to consumers what is in the cosmetics they use every day, and if the contents meet legal requirements.

Students learn to:
- tackle scientific problems by applying their practical skills and their knowledge and understanding of different kinds of analyses
- observe, measure and analyse data collected from different kinds of analyses and evaluate these
- make a plan for the tasks
- calculate the cost of the determinations of ethanol, bearing in mind the time needed, chemicals used and depreciation (write off) of equipment (see example *Report* form in *Your findings*)
- work with others and manage relationships with people
- communicate by written report with consumers.

Developing students' skills
Each project group should consist of three to five students.

Project groups meetings are an opportunity for a professional discussion of progress.

One of the students should keep a booklet with all (half) products and results from the members of his/her group. At the end of the project, the products and results are discussed with the teacher.

Besides the laboratory journal, the written end report, presentation and/or products, the group also writes about how the project was run and everyone's attitude during the project. Filling in a monitor-like questionnaire will help to make the level of commitment clear (see *Student self assessment*).

The teacher guides the group, approves and decides on the plan, distribution of tasks etc.
He/she supports the project group to function independently, and assists the students to learn from each other.
He/she guides the working process and should be approachable for practical questions.

At the beginning, the teacher should set targets and deadlines and help the team with working strategies through appropriate questioning. Let the students write up the minutes and then use these to discuss their progress. If necessary, guide the students in the process.

Students may also need help in estimating for budget management.

Students will find budget management easier if they use a programme such as *Microsoft Excel*.

They use two identical worksheets, one for their estimate and the second for the actual amounts and hours.

They should compare the theoretical with the actual costs (see *Your findings* in the Student's document).

Budget management example for the determination of chloride in cosmetics:

Chemicals	**Price €** / **L**	**Used quantity L**	**Cost €**
nitric acid, concentrated	16.70	7×10^{-3}	0.12
acetone	9.60	5×10^{-2}	0.48
silver nitrate solution	64.70	5×10^{-2}	3.24
Hire	**Price € / hour**	**Time used minutes**	**Costs €**
apparatus	50.00	360	300.00
building (including water, electricity, heating)	200.00	360	1200.00
management time	75.00	120	150.00
analysis time	50.00	360	300.00
Report (writing)	50.00	120	100.00
presentation of Report	50.00	45	37.50
Total costs			2091.34

Organising and managing the class
Students should have experience of potentiometry, iodometry, acid-base titration, precipitation titration, paper chromatography, TLC, GLC and gravimetric analysis.

It is suggested that students work in groups of 3-5, preferably two groups or more.
Every student should do his or her part in planning, practical work and the report.

Students need to recognise the importance of taking care and pride in their work. They need to be gently reminded of this while they are working. During work in the laboratory they may need three types of help:

- help with technical details, including manipulative skills and correct handling of chemicals and equipment
- help with, and vigilance over, health and safety issues
- help with working relations in their group.

Session plan for teachers
Due to the long waiting times about a week will be needed to accomplish this activity.

A suggested approach is as follows:

- Teacher's introduction to the activity (60 minutes)

Outline of the purpose of the project; the students choose a few experiments from the project, after drawing up an inventory of the ingredients in the cosmetics available; statement of expected output (content, format)

- Students arranged into groups
- Collect information and diagnostic assessment with questions related to the subject (120 minutes)
- Carry out practical work (720 minutes)
- Evaluate results, write final report and present the results in a report for consumers (180 minutes)

Technical notes

See the methods on the student sheets.

- **Preparation**

Preparation of solutions
Notes on the preparation of reagent solutions are given in each student sheet.

N.B. Standardisation of titrants: students have to look up an applicable procedure in a textbook and perform the standardisation themselves.

- **Equipment and materials**

Materials and their CAS numbers

Name	**Grade**	**CAS-No.**
Determination of ammonia		
Methanol	Reagent	67-56-1
Barium chloride dihydrate	Reagent	10326-27-9
Boric acid	AR	10043-35-3
Ethanol	Reagent	64-17-5
Sulfuric acid	Reagent	7664-93-9
Sodium hydroxide	Reagent	1310-73-2
Methyl red	Reagent	845-10-3
Methylene blue	Reagent	61-73-4
Determination of chloride		
Nitric acid, concentrated	Reagent	7697-37-2
Acetone	Reagent	67-64-1
Silver nitrate	AR	7761-88-8
Identification of oxidising agents		
Sodium persulfate	Reagent	7775-27-1
Potassium persulfate	Reagent	7727-21-1
Ammonium persulfate	Reagent	7727-54-0
Potassium bromate	Reagent	7758-01-2
Sodium bromate	Reagent	7789-38-0
Potassium iodide	Reagent	7681-11-0
Ethanol absolute	Reagent	64-17-5
Methanol	Reagent	67-56-1
Toluene	Reagent	108-88-3
3-methyl butan-1-ol	Reagent	123-51-3
Starch	Reagent	9005-84-9
Hydrochloric acid, concentrated	Reagent	7647-01-0
Hydrogen peroxide	Reagent	7722-84-1
Determination of hydrogen peroxide		
Sulfuric acid, concentrated	Reagent	7664-93-9
Potassium iodide	Reagent	7681-11-0
Ammonium molybdate	Reagent	11098-84-3
Sodium thiosulfate	AR	7772-98-7
Starch	Reagent	9005-84-9

Identification of chlorates of the alkali metals

Potassium chlorate	Reagent	3811-04-9
Potassium bromate	Reagent	7758-01-2
Potassium iodide	Reagent	7681-11-0
Ammonia, concentrated	Reagent	7664-41-7
Acetone	Reagent	67-64-1
Butanol	Reagent	71-36-3
Hydrochloric acid, concentrated	Reagent	7647-01-0
Starch	Reagent	9005-25-8

Determination of chlorates of the alkali metals

Zinc powder	Reagent	7440-66-6
Silver nitrate	Reagent	7761-88-8
Acetic acid, concentrated	Reagent	64-19-7

Identification of hydrogen sulfites

Hydrochloric acid	Reagent	7647-01-0
Potassium iodate starch paper		

Determination of hydrogen sulfites

Potassium iodate	Reagent	7758-05-6
Hydrogen peroxide	Reagent	7722-84-1
Sodium hydroxide	Reagent	1310-732
Phosphoric acid, concentrated	Reagent	7664-38-2
Methanol	Reagent	67-56-1
Ethanol	Reagent	64-17-5
Methyl red	Reagent	493-52-7
Methylene blue	Reagent	28983-56-4
Nitrogen	Reagent	7727-37-9

Identification of free sodium and potassium hydroxides

Disodium tetra borate decahydrate	AR	1303-96-4

Determination of free sodium and potassium hydroxides

Titration in aqueous medium

Hydrochloric acid, concentrated	Reagent	7647-01-0

Titration in propane-2-ol

Hydrochloric acid, concentrated	Reagent	7647-01-0
Propane-2-ol	Reagent	67-63-0

Determination of methanol in relation to ethanol or propane-2-ol

Methanol	AR	67-56-1
Ethanol, absolute	AR	64-17-5
Propane-2-ol	AR	67-63-0

Determination of zinc

2-methyl-8-quinolinol	Reagent	826-81-3
Ammonium acetate	Reagent	631-61-8
Glacial acetic acid	Reagent	64-19-7
Ammonia, concentrated	Reagent	7664-41-7

Equipment

Determination of ammonia

- centrifuge with stoppered 100 mL bottles
- steam distillation apparatus
- pH-millivolt meter with glass and reference (Ag/AgCl/Cl^-) electrode
- usual laboratory glassware and equipment

Determination of chloride

- heater with magnetic stirrer
- pH/millivolt meter suitable for potentiometric titration, with silver electrode and calomel reference electrode
- usual laboratory glassware and equipment

Identification of oxidising agents

- apparatus for descending paper chromatography
- chromatography paper (Whatman paper No 3 and No 4 or their equivalents)
- standard flasks, 100 mL
- micropipette 1 µL
- fluted filters

Determination of hydrogen peroxide

- burette, 50 mL
- usual laboratory glassware

Identification of chlorates of the alkali metals

- normal equipment for thin layer chromatography
- ready-for-use cellulose thin-layer plates (0.25 mm)

Determination of chlorates of the alkali metals

- centrifuge
- pH/millivolt meter suitable for potentiometric titration
- silver electrode and calomel reference electrode
- normal laboratory equipment

Identification of hydrogen sulfites

- flask (25 mL) fitted with a short reflux condenser
- normal laboratory equipment

Determination of hydrogen sulfites

- distillation apparatus
- normal laboratory equipment

Identification of free sodium and potassium hydroxides

- pH/millivolt meter suitable for potentiometric titration
- glass membrane electrode and calomel reference electrode
- usual laboratory glassware

Determination of free sodium and potassium hydroxides

- pH/millivolt meter suitable for potentiometric titration, preferably with recorder
- glass electrode and calomel reference electrode.
- usual laboratory glassware

Determination of methanol in relation to ethanol or propane-2-ol

- gas chromatograph: with polar column Tenax, nitrogen carrier gas and FID detector (capillary column GC will give even better separation)
- volumetric flasks, 100 mL
- pipettes, 2 mL, 20 mL, 0 to 1 mL
- micro syringes 0 to 100 µL and 0 to 5 µL

Determination of zinc

- drying oven regulated to a temperature 150 ± 2 °C
- hotplate
- glass filter crucibles G-4
- vacuum flasks, 500 mL
- water-jet pump
- thermometer graduated from 0 to 100 °C
- desiccator with a suitable desiccant and humidity indicator e.g. silica gel or equivalent
- filter paper, Whatman No 4 or equivalent
- usual laboratory glassware

Testing prior knowledge

Select the correct answer.

1. Determination of ammonia
In case of a back titration you titrate...
a. potentiometrically with a glass-calomel electrode
b. with a mixed indicator
c. an intermediate component and not the component to be measured
d. an excess of reagent

2. Determination of chloride
A reference electrode is...
a. a conductor in the solution to be titrated
b. a half-cell with a constant potential
c. a platinum wire
d. a glass bulb containing diluted hydrochloric acid

3. Identification of oxidising agents and determination of hydrogen peroxide in hair-care products
The problem of adding the starch solution too early:
a. I_2 oxidises the starch
b. I_2 adsorbs on(to) the starch
c. I_2 reduces the starch
d. I_2 is hydrolyses the starch

4. Identification and determination of chlorates of the alkali metals
The titration curve of the titration of chloride with Ag^+ is:
a. a typical S curve whereas the potential in the equivalent point descends
b. a typical S curve whereas the potential in the equivalent point ascends
c. a V shape where the potential before the equivalent point descends and after it, ascends
d. a V shape where the potential before the equivalent point ascends and after it, descends

5. Identification and determination of hydrogen sulfites
What is the pH of the solution at the equivalent point?
a. 4.8
b. 8.5
c. 7.0
d. It depends of the concentration of the acid in the solution

6. Determination and identification of free sodium and potassium hydroxide
When, instead of NaOH, you calculate the content as percent by mass KOH, then the outcome would be:
a. a smaller number
b. an equal number
c. a larger number
d. not enough data

7. Determination of methanol in relation to ethanol or propan-2-ol (non-aerosol samples)

The procedure uses ethanol or propan-2-ol for the internal standard.
The biggest advantage of using an internal standard would be:

a. less disturbance by variation in injected volume
b. less work using pipettes
c. higher sensitivity
d. there is no advantage

8. Determination of zinc

Why is a desiccator used for cooling?

a. to prevent the adsorption of water vapour from the air
b. to prevent reaction of the precipitate with oxygen from the air
c. to absorb any remaining water out of the precipitate
d. to slow down the rate of cooling

Note:
Answers are given in red. These need to be removed before the test is used.

Testing prior knowledge

Select the correct answer.

1. Determination of ammonia
In case of a back titration you titrate...
a. potentiometrically with a glass-calomel electrode
b. with a mixed indicator
c. an intermediate component and not the component to be measured
d. an excess of reagent

2. Determination of chloride
A reference electrode is...
a. a conductor in the solution to be titrated
b. a half-cell with a constant potential
c. a platinum wire
d. a glass bulb containing diluted hydrochloric acid

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c. a V shape where the potential before the equivalent point descends and after it, ascends
d. a V shape where the potential before the equivalent point ascends and after it, descends

5. Identification and determination of hydrogen sulfites
What is the pH of the solution at the equivalent point?
a. 4.8
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d. It depends of the concentration of the acid in the solution

6. Determination and identification of free sodium and potassium hydroxide
When, instead of NaOH, you calculate the content as percent by mass KOH, then the outcome would be:
a. a smaller number
b. an equal number
c. a larger number
d. not enough data

7. Determination of methanol in relation to ethanol or propan-2-ol (non-aerosol samples)

The procedure uses ethanol or propan-2-ol for the internal standard.
The biggest advantage of using an internal standard would be:
a. less disturbance by variation in injected volume
b. less work using pipettes
c. higher sensitivity
d. there is no advantage

8. Determination of zinc

Why is a desiccator used for cooling?
a. to prevent the adsorption of water vapour from the air
b. to prevent reaction of the precipitate with oxygen from the air
c. to absorb any remaining water out of the precipitate
d. to slow down the rate of cooling