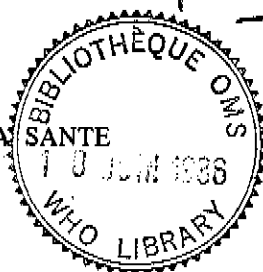




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BASIC TESTS FOR PHARMACEUTICAL DOSAGE FORMS

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Drug preparations - methods

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1. INTRODUCTION

The programme of Basic Tests is discussed in detail in a number of reports of the WHO Expert Committee on Specifications for Pharmaceutical Preparations¹⁻³ as well as in the manual on "Basic tests for pharmaceutical substances".⁴ This document complements this manual, in that it contains identity tests for 85 of these drugs in the finished pharmaceutical dosage forms: tablets, capsules, injections, drugs, solutions, suspensions, creams, lotions. Further tests are in preparation.

The proposed tests are preceded by a short description of the minimum facilities and equipment needed to carry out the work, and basic rules for sampling and inspection of dosage forms are also briefly described.

¹WHO Technical Report Series, No. 645, 1980

²WHO " " " , No. 681, 1982

³WHO " " " , No. 704, 1984

⁴Basic tests for pharmaceutical substances, World Health Organization, Geneva, 1986.

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Each of the tests described has been verified in at least four laboratories in different countries. Further validation of the tests (see Annex) on locally available samples are invited. These results or any other relevant comments should be forwarded to:

Pharmaceuticals Unit
World Health Organization,
1211 Geneva 27,
Switzerland

Most of the required reagents are described in the manual on "Basic tests for pharmaceutical substances." The small number of additional reagents needed for testing dosage forms are listed at the end of the document.

2. RECOMMENDED FACILITIES

A full pharmacopoeial analysis of a drug substance or drug product can only be performed in an adequately equipped drug quality control laboratory. Recommendations are contained in the 29th WHO Expert Committee report on the design and organization of a modest control laboratory.

Provision of the facilities to undertake basic tests could be regarded as the first step toward the development of this recommended laboratory.

2.1 Generalities

The work place should be well illuminated by indirect natural light. It should be air-conditioned or heated, according to the climate. In the latter case a closed convector heater or radiator should be chosen that can be operated without fire hazard. The doors should open outwards and all other locally operative fire and safety regulations should be respected.

The floor-covering should be continuous, fire proof, and easily washable. The walls, furniture and the frames of the windows and doors should be easily washable. All these elements should be of a neutral colour (preferably white or pale beige) to facilitate the evaluation of colour reactions.

2.2 Furniture

Benches should be placed against a wall and they should receive natural light from the left side. A fume cupboard, or a table with a ventilator above it should be installed in one corner of the room.

The top of working benches should have a white or black acid-resistant plastic cover; a glass or ceramic top results in increased breakage of glassware. Unpainted wooden surfaces should be impregnated with soft paraffin. Paints or resins used to protect these surfaces should be resistant to dilute acids. The height of the working bench should enable work to be undertaken comfortably in both standing and sitting positions. The benches should include shelves and drawers that are not unnecessarily deep for the items that will be stored in them. Chairs should be strongly built and stable.

Cupboards and glass cases are preferable for many purposes because they can easily be reached and cleaned. Reagent shelves should be covered with unpolished glass plates and firmly fixed to walls or benches. Flammable solvents and concentrated mineral acids should be stored in well-closed containers of no more than 2 litres capacity. Larger volumes held in stock should be stored in a separate room that complies with locally-operative fire safety requirements. Concentrated acids and ammonia solutions should be stored separately, preferably in a fume cupboard. If they are stored elsewhere the cupboard should be recessed in a wall, lined with tiles or acid-resistant plastic, and fitted with a glass door in an acid-resistant plastic frame.

2.3 Electric lighting and power, gas and water supply, ventilation

All electrical circuits, gas and water supplies must comply with locally-operative safety standards.

Artificial lighting should not cast shadow and all fittings should have a protective cover that is resistant to acids and solvents.

All electrical sockets should be fitted with automatic fuses.

If no gas supply is available, spirit lamps or electric heaters can be used. Electric heaters should be equipped with heating-output regulators and the heating coil must be fully insulated. Flammable liquids must be heated in a water-bath.

Working benches should be equipped with a water tap and a sink. Taps should be fitted with three bib valves one of which is connected to the water-pump. The waste water pipes must be resistant to acids and solvents.

To avoid contamination of distilled water by acid or ammonia fumes the distillation apparatus and the ion-exchanger must be sited well away from working areas.

Good ventilation must be ensured. Windows should open freely if there is no air-conditioning.

2.4 Equipment

Basic tests require little laboratory equipment. Test tubes are used for the majority of the tests. In some cases porcelain crucibles are also needed. Flasks and beakers are necessary for the preparation of reagents.

Glassware and porcelain dishes

test-tubes
porcelain or quartz crucibles
flasks, flat or round bottomed
wash bottles, glass or plastic
evaporating dishes
crystallizing dishes
watch-glasses
beakers
mortar with pestle
filter funnels
separatory funnels
sintered glass funnels
bottles with screw-cap, wide-mouthed

Volumetric measuring vessels

measuring cylinders
pipettes, graduated
dropping pipettes
volumetric flasks

Other equipment

sampling spoons
wooden test-tube holder
test-tube brush
test-tube clamp
crucible tongue
suction bottle
water-pump
water-bath
oven
burners, Bunsen or alcohol burner
asbestos gauze
tripod
distillation apparatus (Liebig-type condensers)
desiccator
melting point apparatus
glass capillaries
magnesia sticks
platinum wire sealed to a glass rod
filter-paper
pH-indicator paper
polyethylene dropping bottles
UV-light source
balance (precision 10 mg)

3. SAMPLING AND INSPECTION

Recommendations on sampling of materials to be tested are contained in the 30th Expert Committee on Specifications for Pharmaceutical preparations (WHO Technical Report Series, in print).

Visual inspection of samples is vitally important. Rejection often results from changes to the physical appearance of dosage forms or defects in packaging and labelling.

Drugs with inadequate, damaged or missing labels should, in most cases, be referred to a fully equipped drug control laboratory.

The label should be checked to ensure that it provides the following information: the name of the drug; its strength, potency or concentration; the name of the manufacturer, the batch or lot number and the expiry date. Drugs repackaged away from the original manufacturing premises should bear the control number of the responsible analytical laboratory.

Commonly encountered defects in physical characteristics include:

-for tablets

excessive powder and/or pieces of tablets at the bottom of the container (from abraded, crushed or broken tablets);
cracks or chips in the tablet surfaces or coatings, swelling, mottling, discoloration, fusion of tablets;
appearance of crystals on the container walls or on the tablets.

-for capsules

hardening or softening, cracking, swelling, mottling or discoloration of the shell etc.

4. DRUG INDEX

acetazolamide tablets
acetylsalicylic acid tablets
aluminium hydroxide tablets
aminophylline injection
aminophylline tablets
amitriptyline hydrochloride tablets
amodiaquine hydrochloride tablets
ampicillin capsules
ampicillin sodium powder for injection
ascorbic acid tablets
atropine sulfate injection
azathioprine tablets

benzyl benzoate lotion
benzylpenicillin potassium powder for injection
benzylpenicillin sodium powder for injection
betamethasone valerate cream
busulfan tablets

carbamazepine tablets
chlorambucil tablets
chloramphenicol capsules
chloramphenicol palmitate oral suspension
chloroquine phosphate tablets
chloroquine sulfate tablets
chlorphenamine hydrogen maleate tablets
chlorpromazine hydrochloride tablets
clomifene citrate tablets
codeine phosphate tablets
colchicine tablets
cyclophosphamide tablets
cytarabine injection

dapsone tablets
dexamethasone tablets
diazepam tablets
diethylcarbamazine dihydrogen citrate tablets
digoxin tablets

ephedrine hydrochloride tablets
epinephrine hydrochloride ophthalmic solution
ergotamine tartrate tablets
ethambutol hydrochloride tablets
ethinylestradiol tablets

ferrous sulfate tablets
folic acid tablets
furosemide injection
furosemide tablets

glucose injection
griseofulvin tablets

homatropine hydrobromide ophthalmic solution

ibuprofen tablets
indometacin capsules
iodine solution
isoniazid tablets
isoprenaline sulfate tablets

levodopa tablets
lidocaine hydrochloride injection

mannitol injection
mebendazole tablets
methyldopa tablets
metronidazole tablets

neomycin sulfate tablets
nitrofurantoin tablets
nystatin tablets

paracetamol tablets
phenobarbital tablets
prednisolone tablets

primaquine diphosphate tablets
procainamide hydrochloride tablets
promethazine hydrochloride injection
promethazine hydrochloride tablets
propranolol hydrochloride tablets
pyridoxine hydrochloride tablets

quinidine sulfate tablets

reserpine tablets
retinol oral solution
riboflavin tablets

salbutamol sulfate tablets
salicylic acid lotion
sodium citrate solution
sodium hydrogen carbonate tablets
sodium nitrite injection
streptomycin sulfate powder for injection
sulfacetamide sodium ophthalmic solution
sulfadimidine tablets

tetracycline hydrochloride capsules
thiamine hydrochloride tablets
trimethoprim tablets

5. TEST PROCEDURES

ACETAZOLAMIDE TABLETS

Description. Each tablet usually contains 250 mg of acetazolamide.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 30 mg and 40 mg of acetazolamide.
2. Grind the tablets, weigh out the above calculated equivalent amounts to acetazolamide as powdered material and use them directly: 30 mg for test substance 1; 40 mg for test substance 2.

IDENTITY TESTS

Colour and other reactions

1. Transfer test substance 1 to a test-tube, add 2.0 ml of hydrochloric acid (~70 g/l)TS and 0.05 g of zinc R powder; an odour of hydrogen sulfide is perceptible. Place a strip of lead acetate paper R or lead nitrate paper R above the tube; the colour of the paper turns brown-black.
2. To test substance 2 add 5 ml of water, about 0.1 ml of sodium hydroxide (~80 g/l)TS and shake. Following this add about 0.1 ml of copper(II) sulfate (160 g/l)TS; a blue-green precipitate is produced.

ACETYLSALICYLIC ACID TABLETS

Description. Each tablet usually contains 100-500 mg of acetylsalicylic acid.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 20 mg of acetylsalicylic acid.
2. Grind the tablets, weigh out the above calculated equivalent amount to acetylsalicylic acid as powdered material and use it directly as the test substance.

IDENTITY TESTS

Heating behaviour. Heat a small quantity of the test substance in a test-tube; the melt has a strong odour of acetic acid. On further heating, the colour of the melt changes from yellow to brown and then to black.

Colour and other reactions

1. Transfer half the quantity of the test substance to a suitable white test plate or a watch-glass placed on a white background, and add 1 drop of ferric chloride (25 g/l)TS; no violet colour is produced.
2. Place the remaining test substance on a suitable white test plate or a watch-glass placed on a white background, and add 1 drop of potassium hydroxide/ethanol TS. After 1 minute add 1 drop of ferric chloride (25 g/l)TS; a violet colour is produced.

ALUMINIUM HYDROXIDE TABLETS

Description. Each tablet usually contains 500 mg of aluminium hydroxide.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 0.15 g and 0.7 g of aluminium hydroxide.
2. Grind the tablets, weigh out the above calculated equivalent amount to aluminium hydroxide as powdered material and use it directly: 0.15 g for test substance 1; 0.7 g for test substance 2.

IDENTITY TESTS

Colour and other reactions

1. To test substance 1 add 5 ml of sodium hydroxide (~80 g/l)TS, heat to boiling and filter. To the filtrate add 0.5 g of ammonium chloride R and shake; a white, gelatinous precipitate is gradually produced.
2. Shake test substance 2 with 10 ml of freshly boiled and cooled water for 1 minute, and filter; the filtrate is neutral when tested with pH-indicator paper R.

AMINOPHYLLINE INJECTION

Description. The injection is a sterile solution usually containing 25 mg of aminophylline in 1.0 ml of a suitable vehicle.

Preparation of the sample

Pool the contents of the ampoules equivalent to 0.25 g of aminophylline and use it directly as the test solution. Divide the test solution into two equal volumes.

IDENTITY TESTS

Colour and other reactions

1. To 1 volume of the test solution add 6 ml of water and, drop by drop while shaking, about 4 ml of hydrochloric acid (~70 g/l)TS until acidic. Collect the precipitate on a filter, wash it with water and dry it at 105 °C for 1 hour; melting behaviour, about 270 °C (keep the residue for test 2).
2. Place 10 mg of the residue obtained in test 1 on a porcelain dish, add 1.0 ml of hydrochloric acid (~250 g/l)TS and about 0.5 ml of hydrogen peroxide (~330 g/l)TS. Evaporate to dryness of a water-bath. To the dried orange coloured residue add 1 drop of ammonia (~100 g/l)TS; the colour changes to purple and it is destroyed by the addition of a few drops of sodium hydroxide (~80 g/l)TS.
3. To the remaining volume of the test solution add 0.5 ml of sodium hydroxide (~80 g/l)TS and 0.25 ml of copper(II) acetate (45 g/l)TS; a violet colour is produced. Add 0.5 ml of potassium-mercuric iodide TS; a white precipitate is produced, which rapidly changes to a violet colour.

AMINOPHYLLINE TABLETS

Description. Each tablet usually contains 100-200 mg of aminophylline.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.5 g of aminophylline.
2. Grind the tablets, weigh out the above calculated equivalent amount to aminophylline as powdered material, shake it with 25 ml of water, filter, to the filtrate add 1.0 ml of hydrochloric acid (~70 g/l)TS, stir and chill if necessary, to precipitate the theophylline. Filter and retain the filtrate free from washings, to be used as the test solution. Wash the precipitate on the filter with ice-water, dry it at 150 °C for 1 hour and use the dried material as the test substance.

IDENTITY TESTS

Colour and other reactions

1. To 10 mg of the test substance, contained in a porcelain dish, add 1.0 ml of hydrochloric acid (~250 g/l)TS and about 0.5 ml of hydrogen peroxide (~330 g/l)TS and evaporate to dryness on a water-bath. Add 1 drop of

ammonia (~100 g/l)TS to the residue; a purple colour is produced which is destroyed by the addition of a few drops of sodium hydroxide (~80 g/l)TS.

2. Prepare a saturated solution with a portion of the test substance in water and add a few drops of tannic acid (100 g/l)TS; a precipitate is produced which is soluble in an excess of the reagent.

3. To 5 ml of the test solution add 0.5 ml of sodium hydroxide (~80 g/l)TS and 5 drops of copper(II) acetate (45 g/l)TS; a violet colour is produced. Add 0.5 ml of potassium-mercuric iodide TS; a white precipitate is produced, which rapidly changes to a violet colour.

AMITRIPTYLINE HYDROCHLORIDE TABLETS

Description. Each tablet usually contains 25 mg of amitriptyline hydrochloride. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amounts equivalent to 10 mg and 0.10 g of amitriptyline hydrochloride.
2. Grind the tablets or cores, weigh out the above calculated equivalent amounts to amitriptyline hydrochloride as powdered material and use 10 mg directly for test substance 1. For test substance 2, shake 0.10 g of the equivalent amount of powder with 5 ml of hydrochloric acid (0.05 mol/l)VS for 30 minutes. Add 5 ml of water and 1.0 g of sodium chloride R, shake it for 3 minutes and make alkaline with sodium hydroxide (~80 g/l)TS. Extract with 3 quantities, each of 10 ml of ether R, separate the ether layers carefully and filter. Extract the combined filtrate with 5 ml of hydrochloric acid (0.05 mol/l)VS, separate the acid solution and evaporate it to dryness on a water-bath. Dissolve the residue in 5 ml of chloroform R, filter, evaporate the filtrate on a water-bath and use the residue. For the test solution, shake 0.10 g of the equivalent amount of powder with 5 ml of water, filter and use the filtrate.

IDENTITY TESTS

Melting point. Test substance 2 melts at about 194 °C.

Colour and other reactions

1. To test substance 1 add about 2 ml of sulfuric acid (~1760 g/l)TS; an orange-red colour is produced. Add 0.1 ml of formaldehyde TS; the colour turns to brown.
2. To the test solution add 0.10 ml of nitric acid (~130 g/l)TS; a white precipitate which may appear dissolves on stirring. Check the solution with pH-indicator paper R to assure that it is acidic and add 2.0 ml of silver nitrate (40 g/l)TS; a white, curdy precipitate is produced.

AMODIAQUINE HYDROCHLORIDE TABLETS

Description. Each tablet usually contains 200 mg of amodiaquine dihydrochloride.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.75 g of amodiaquine dihydrochloride.
2. Grind the tablets, weigh out the above calculated equivalent amount to amodiaquine dihydrochloride as powdered material and use it directly as test substance 1. Divide the test substance into 3 equal parts.
3. Shake 1 part of test substance 1 with 20 ml of ethanol (~750 g/l)TS, filter, evaporate the filtrate to dryness, recrystallize from dehydrated ethanol R, and dry to constant weight at 105 °C. Use the crystals as test substance 2.
4. Suspend 1 part of test substance 1 in 5 ml of water. Place a strip of filter-paper into the suspension and allow the solution to ascend for about 4 cm. Take out the strip, cut away the lower dipped portion as well as the part that has not been wetted by the solution and dry the remaining part of the strip in air at room temperature (test-paper).

IDENTITY TESTS

Melting behaviour. Test substance 2 melts at about 160 °C with slight evolution of gas.

Colour and other reactions

1. Shake the remaining part of test substance 1 with 50 ml of water and filter. Make the filtrate alkaline with about 1 ml of ammonia (~100 g/l)TS and allow to stand for 30 minutes. Filter, wash the filter with water until free from chlorides. Dry the residue remaining on the filter at 105 °C for 2 hours; it melts at about 202 °C with decomposition and discoloration.
2. To 0.10 g of test substance 2 add 2 ml of nitric acid (~1000 g/l)TS; a red solution is produced.

Alternate test by filter-paper technique:

Place onto the test-paper 1 drop of nitric acid (~1000 g/l)TS; an orange coloured spot is produced.

3. Suspend 0.05 g of test substance 2 with 2.0 ml of water and filter. To the filtrate add a few drops of nitric acid (~130 g/l)TS and 0.5 ml of silver nitrate (40 g/l)TS; a white, curdy precipitate is produced in a yellow supernatant liquid.

AMPICILLIN CAPSULES

Description. Each capsule contains ampicillin or ampicillin trihydrate usually equivalent to 250-500 mg of anhydrous ampicillin.

Preparation of the sample

1. Weigh the contents of 1 capsule and calculate the amounts equivalent to 30 mg and 0.10 g of anhydrous ampicillin.

2. Empty the capsules, weigh out the above calculated equivalent amounts to anhydrous ampicillin and use them directly: 30 mg for test substance 1 and divide it into three equal parts; 0.10 g for test substance 2.
3. Suspend test substance 2 in 5 ml of ethanol (~750 g/l)TS. Place 2 strips of filter-paper into the suspension and allow the solution to ascend for about 4 cm. Take out the strips, cut away the lower dipped portion as well as the part that has not been wetted by the solution and dry the remaining part of the strips in air at room temperature (test-paper).

IDENTITY TESTS

Colour and other reactions

1. Shake 1 part of test substance 1 with 3 ml of water and filter. To the filtrate add 0.10 g of hydroxylamine hydrochloride R, about 0.4 ml of sodium hydroxide (~80 g/l)TS and allow to stand for 5 minutes. Following this add 1.3 ml of hydrochloric acid (~70 g/l)TS and 0.5 ml of ferric chloride (25 g/l)TS; a violet-red to violet-brown colour is produced.

Alternate test by filter-paper technique:

Place onto a test-paper 1 drop of hydroxylamine hydrochloride (10 g/l)TS, followed by a drop of sodium hydroxide (~80 g/l)TS and allow to react for 5 minutes. Apply at the same place on the test-paper 1 drop of hydrochloric acid (~70 g/l)TS and 1 drop of ferric chloride (25 g/l)TS; a violet-red ring is produced.

2. Shake 1 part of test substance 1 with 1.0 ml of water and filter. To the filtrate add 1.0 ml of a solution composed of 2.0 ml of potassio-cupric tartrate TS and 6 ml of water; a red-violet colour is produced, turning green on standing.

Alternate test by filter-paper technique:

Place onto a test-paper 1 drop of a solution composed of 2.0 ml of potassio-cupric tartrate TS and 6 ml of water; a light violet spot is produced.

3. Dissolve 10 mg of triketohydrindene hydrate R in 10 ml of ethanol (~750 g/l)TS and place 0.10 ml on a strip of filter-paper, and dry it at 105 °C. Superimpose the spot with about 0.1 ml of a suspension of the remaining part of the test substance 1 in 10 ml of water, heat at 105 °C for 5 minutes and allow to cool; a violet colour is obtained.

AMPICILLIN SODIUM POWDER FOR INJECTION

Description. Each vial contains a sterile powder of ampicillin sodium usually equivalent to 500 mg of anhydrous ampicillin.

Preparation of the sample

1. Weigh the contents of 1 vial and calculate the amounts equivalent to 0.05 g and 0.10 g of ampicillin sodium.
2. Empty the vials, weigh out the above calculated equivalent amounts to ampicillin sodium and use them directly: 0.05 g for test substance 1 and divide it into five equal parts; 0.10 g for test substance 2.

3. Suspend test substance 2 in 5 ml of ethanol (~750 g/l)TS. Place 2 strips of filter-paper into the suspension and allow the solution to ascend for about 4 cm. Take out the strips, cut away the lower dipped portion as well as the part that has not been wetted by the solution and dry the remaining part of the strips in air at room temperature (test-paper).

IDENTITY TESTS

Colour and other reactions

1. Dissolve half of 1 part of test substance 1 in 3 ml of water, add 0.10 g of hydroxylamine hydrochloride R, 1.0 ml of sodium hydroxide (~80 g/l)TS and allow to stand for 5 minutes. Following this add 1.0 ml of hydrochloric acid (~70 g/l)TS and 0.5 ml of ferric chloride (25 g/l)TS; a violet-red to violet-brown colour is produced.

Alternate test by filter-paper technique:

Place onto a test-paper 1 drop of hydroxylamine hydrochloride (10 g/l)TS, followed by 1 drop of sodium hydroxide (~80 g/l)TS, and allow to react for 5 minutes. Following this apply at the same place on the test-paper 1 drop of hydrochloric acid (~70 g/l)TS and 1 drop of ferric chloride (25 g/l)TS; a violet-red ring is produced.

2. Dissolve 1 part of test substance 1 in 1.0 ml of water and add 1.0 ml of a solution composed of 2.0 ml of potassio-cupric tartrate TS and 6 ml of water; a red-violet colour is produced, turning green on standing.

Alternate test by filter-paper technique:

Place onto a test-paper 1 drop of a mixture composed of 2.0 ml of potassio-cupric tartrate TS and 6 ml of water; a light violet spot is produced.

3. Dissolve 10 mg of triketohydrindene hydrate R in 10 ml of ethanol (~750 g/l)TS and place 0.1 ml on a strip of filter-paper, and dry it at 105 °C. Superimpose the spot with 0.1 ml of a solution of 1 part of test substance 1 in 10 ml of water, heat at 105 °C for 5 minutes and allow to cool; a violet spot is obtained.

4. Dissolve 2 parts of test substance 1 in 2.0 ml of water, acidify with 3-4 drops of glacial acetic acid R, filter and add 1.0 ml of magnesium uranyl acetate TS or zinc uranyl acetate TS to the filtrate. Scratch the inside of the tube with a glass rod to induce crystallization; a yellow, crystalline precipitate is produced.

ASCORBIC ACID TABLETS

Description. Each tablet usually contains 50 mg of ascorbic acid.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.30 g of ascorbic acid.
2. Grind the tablets, weigh out the above calculated equivalent amount to ascorbic acid as powdered material and use it directly as the test substance. Divide the test substance into six equal parts.

3. Shake 4 parts of the test substance with 20 ml of water, filter and use the filtrate as the test solution.
4. Suspend 1 part of the test substance in 10 ml of ethanol (~750 g/l)TS. Place 3 strips of filter-paper into the suspension and allow the solution to ascend for about 4 cm. Take out the strips, cut away the lower dipped portion as well as the part that has not been wetted by the solution, and dry the remaining part of the strip in air at room temperature (test-paper).

IDENTITY TESTS

Heating behaviour. Heat a small quantity of the test substance in a test-tube; it melts, acquires a brown colour and has an odour resembling caramel. Ignite the melt; it swells and burns.

Colour and other reactions

1. To 2.0 ml of the test solution add 1.0 g of sodium hydrogen carbonate R and 20 mg of ferrous sulfate R; a violet colour is produced. Add 2.0 ml of hydrochloric acid (~70 g/l)TS; the colour disappears.

Alternate test by filter-paper technique:

Place onto a test-paper 1 small drop of sodium hydrogen carbonate (40 g/l)TS, followed by 1 drop of ferrous sulfate (15 g/l)TS; a violet spot is produced. Then apply at the same place on the test-paper 1 drop of hydrochloric acid (~70 g/l)TS; the spot disappears.

2. To 2.0 ml of the test solution add 0.5 ml of potassium permanganate (10 g/l)TS; the initial violet colour is immediately discharged but a slight brown precipitate may appear.

Alternate test by filter-paper technique:

Place onto a test-paper 1 drop of potassium permanganate (10 g/l)TS; the violet colour is discharged but a brown spot appears.

3. To 2.0 ml of the test solution add 2-3 drops of silver nitrate (40 g/l)TS; a dark grey precipitate is produced.

Alternate test by filter-paper technique:

Place onto a test-paper 1 drop of silver nitrate (40 g/l)TS; a dark grey spot is produced.

ATROPINE SULFATE INJECTION

Description. The injection is a sterile solution usually containing 0.5-1.0 mg of atropine sulfate in 1.0 ml of a suitable vehicle.

Preparation of the sample

Pool the contents of the ampoules equivalent to 20 mg of atropine sulfate, if necessary, reduce the volume to about 25 ml on a water-bath or dilute it to 25 ml with water and use it directly as the test solution. Divide the test solution into two equal volumes.

IDENTITY TESTS

Colour and other reactions

1. Evaporate 1 volume of the test solution to dryness on a water-bath. To the cooled, white residue add 5 ml of dehydrated ethanol R, shake and filter. Evaporate the filtrate to dryness on a water-bath, to the residue add 3 drops of nitric acid (~1000 g/l)TS and again evaporate to dryness. Cool the residue and add 4 drops of potassium hydroxide/ethanol TS; a brownish violet colour is produced, which slowly disappears and a yellow residue remains.
2. To the second volume of the test solution add a few drops of hydrochloric acid (~70g/l)TS and a few drops of barium chloride (50g/l)TS; a white precipitate is produced.

AZATHIOPRINE TABLETS

Description. Each tablet usually contains 50 mg of azathioprine.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.075 g of azathioprine.
2. Grind the tablets, weigh out the above calculated equivalent amount to azathioprine as powdered material and use it directly as the test substance. Divide the test substance into three equal parts.

IDENTITY TESTS

Colour and other reactions

1. Heat 1 part of the test substance with 100 ml of water and filter. To 5 ml of the filtrate add 1 ml of hydrochloric acid (~250 g/l)TS and 10 mg of zinc R powder, and allow to stand for 5 minutes; the solution becomes yellow. Filter, cool in ice, add a few drops of sodium nitrite (10 g/l)TS and a few drops of sulfamic acid (100 g/l)TS. Shake until the bubbles disappear. Add 1.0 ml of 2-naphthol TS; a pale pink precipitate is produced.
2. Fuse 2 parts of the test substance with 0.05 g of potassium nitrate R and 0.10 g of potassium hydroxide R. Cool, dissolve the residue in 20 ml of water and filter. To 5 ml of the filtrate add 1.5 ml of hydrochloric acid (~70 g/l)TS and 0.5 ml of barium chloride (50 g/l)TS; a white turbidity is produced.

BENZYL BENZOATE LOTION

Description. The lotion usually contains 250 mg of benzyl benzoate suspended or dispersed in 1.0 ml of a suitable vehicle.

Preparation of the sample

Dissolve a volume of the lotion equivalent to 2.5 g of benzyl benzoate in 25 ml of ethanol (~750 g/l)TS and filter. Add 10 ml of sodium hydroxide (~80 g/l)TS and evaporate the ethanol on a water-bath. Cool the

solution and shake it with 15 ml of ethyl acetate R (keep the aqueous layer as the test solution). Wash the ethyl acetate layer with 10 ml of sodium hydroxide (~80 g/l)TS, filter and evaporate to a low volume on a water-bath. Use the oily colourless liquid as the test liquid.

IDENTITY TESTS

Heating behaviour. Heat cautiously a few drops of the test liquid; it smokes, bursts into flame and burns with a rather sooty flame.

Colour and other reactions

1. To 1-2 drops of the test liquid add 5 ml of sodium carbonate (50 g/l)TS and 5 drops of potassium permanganate (10 g/l)TS; an odour of benzaldehyde is perceptible.
2. Neutralize the test solution with hydrochloric acid (~70 g/l)TS and add 1.0 ml of ferric chloride (25 g/l)TS; a reddish brown precipitate is formed. Shake the precipitous mixture with 10 ml of hydrochloric acid (~70 g/l)TS; a voluminous white precipitate is formed.

BENZYLPENICILLIN POTASSIUM POWDER FOR INJECTION

Description. Each vial contains a sterile powder usually equivalent to 0.6-3.0 g (1-5 million IU) of benzylpenicillin potassium.

Preparation of the sample

1. Weigh the contents of 1 vial and calculate the amounts equivalent to about 5 mg, 0.04 g and 0.10 g of benzylpenicillin potassium.
2. Empty the vials, weigh out the above calculated equivalent amounts to benzylpenicillin potassium and use them directly: about 5 mg for test substance 1; 0.04 g for test substance 2 and divide it into four equal parts; 0.10 g for test substance 3.
3. Suspend test substance 3 in 5 ml of ethanol (~750 g/l)TS, place a strip of filter-paper into the suspension and allow the solution to ascend for about 4 cm. Take out the strip, cut away the lower dipped portion as well as the part that has not been wetted by the solution and dry the remaining part of the strip in air at room temperature (test-paper).

IDENTITY TESTS

Colour and other reactions

1. Dissolve 1 part of test substance 2 in 3 ml of water, add 0.10 g of hydroxylamine hydrochloride R, 1.0 ml of sodium hydroxide (~80 g/l)TS, and allow to stand for 5 minutes. Following this add 1.3 ml of hydrochloric acid (~70 g/l)TS and 0.5 ml of ferric chloride (25 g/l)TS; a violet-red colour is produced.

Alternate test by filter-paper technique:

Place onto the test-paper 1 drop of hydroxylamine hydrochloride (10 g/l)TS, followed by 1 drop of sodium hydroxide (~80 g/l)TS and allow to react for 5 minutes. Following this apply at the same place on the test-paper 1 drop of hydrochloric acid (~70 g/l)TS and 1 drop of ferric chloride (25 g/l)TS; a violet-red ring is produced.

2. Dissolve 1 part of test substance 2 in a few drops of ethanol (~750 g/l)TS, add 1.0 ml of water and 2 drops of ferric chloride (25 g/l)TS; a yellowish precipitate is produced.
3. To 10 mg of paraformaldehyde R dissolved in about 1 ml of sulfuric acid (~1760 g/l)TS add a few crystals of test substance 1; a colourless solution is produced. Heat the solution in a water-bath for 2 minutes and cool; the colour changes to brown-red.
4. Dissolve the remaining parts of test substance 2 in 2.0 ml of water, acidify with 2-4 drops of glacial acetic acid R, and add 1.0 ml of sodium cobaltinitrite (100 g/l)TS; an orange-yellow precipitate is produced.

BENZYL PENICILLIN SODIUM POWDER FOR INJECTION

Description. Each vial contains a sterile powder usually equivalent to 0.6-3.0 g (1-5 million IU) of benzylpenicillin sodium.

Preparation of the sample

1. Weigh the contents of 1 vial and calculate the amounts equivalent to about 25 mg, 0.04 g and 0.10 g of benzylpenicillin sodium.
2. Empty the vials, weigh out the above calculated equivalent amounts to benzylpenicillin sodium and use them directly: 25 mg for test substance 1 and divide it into five equal parts; 0.04 g for test substance 2; 0.10 g for test substance 3.
3. Suspend test substance 3 in 5 ml of ethanol (~750 g/l)TS, place a strip of filter-paper into the suspension and allow the solution to ascend for about 4 cm. Take out the strip, cut away the lower dipped portion as well as the part that has not been wetted by the solution and dry the remaining part of the strip in air at room temperature (test-paper).

IDENTITY TESTS

Melting behaviour. A few crystals of the test substance melt at about 228 °C with decomposition (turns black).

Colour and other reactions

1. Dissolve test substance 1 in 3 ml of water, add 0.10 g of hydroxylamine hydrochloride R, about 0.4 ml of sodium hydroxide (~80 g/l)TS and allow to stand for 5 minutes. Following this add 1.3 ml of hydrochloric acid (~70 g/l)TS and 0.5 ml of ferric chloride (25 g/l)TS; a violet-red colour is produced.

Alternate test by filter-paper technique:

Place onto the test-paper 1 drop of hydroxylamine hydrochloride (10 g/l)TS, followed by 1 drop of sodium hydroxide (~80 g/l)TS and allow to react for 5 minutes. Following this apply at the same place on the test-paper 1 drop of hydrochloric acid (~70 g/l)TS and 1 drop of ferric chloride (25 g/l)TS; a violet-red ring is produced.

2. Dissolve test substance 1 in a few drops of ethanol (~750 g/l)TS, add 1.0 ml of water and 2 drops of ferric chloride (25 g/l)TS; a yellowish precipitate is produced.
3. To 10 mg of paraformaldehyde R dissolved in about 1 ml of sulfuric acid (~1760 g/l)TS add a few crystals of test substance 1; a light yellow colour is produced. Heat the solution in a water-bath for 2 minutes and cool; the colour changes to brown-red.
4. Dissolve test substance 2 in 2.0 ml of water, acidify with 2-4 drops of glacial acetic acid R, filter and to the filtrate add 1.0 ml of magnesium uranyl acetate TS or zinc uranyl acetate TS. Scratch the inside of the tube with a glass rod to induce crystallization; a yellow, crystalline precipitate is produced.

BETAMETHASONE VALERATE CREAM

Description. The cream usually contains 1.2 mg of betamethasone valerate in 1 g of a suitable base equivalent to 1.0 mg of betamethasone per gram.

Preparation of the sample

Withdraw an amount equivalent to 10 mg of betamethasone valerate, shake it well with 30 ml of methanol R and 20 ml of cyclohexane R. Separate the methanol layer and wash it with two 10 ml portions of cyclohexane R. Filter the methanol layer and evaporate the filtrate on a water-bath to a volume of 10 ml and use it as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To 1 ml of formaldehyde/sulfuric acid TS add 2.0 ml of the test solution and shake well; a pink to orange colour is produced. Heat the solution on a water-bath for 1 minute; the colour changes to brown-violet.
2. Add 1.0 ml of hot potassio-cupric tartrate TS to 2.0 ml of the test solution. Warm the solution for 10-15 minutes; the slightly yellowish colour of the initial solution changes gradually to blue and green containing a few specks of reddish-brown precipitate.
3. Mix 2-3 drops of potassium dichromate (~100 g/l)TS with about 0.5 ml of sulfuric acid (~1760 g/l)TS and heat on a water-bath for 5 minutes; the solution wets the sides of the tube. Cool and add drop by drop 3 ml of the test solution. Shake well and heat again for 15 minutes; the colour of the solution turns to violet-black and it no longer wets the sides of the tube.

BUSULFAN TABLETS

Description. Each tablet usually contains 2.0 mg of busulfan. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amount equivalent to 60 mg of busulfan.
2. Grind the tablets or cores, weigh out the above calculated equivalent amount to busulfan as powdered material, shake it with 30 ml of acetone R, filter, evaporate the filtrate to dryness on a water-bath, crystallize from a small volume of acetone R, separate the crystals, dry them at 80 °C for 2 hours and use this material as the test substance.

IDENTITY TESTS

Melting point. The test substance melts at about 117 °C.

Colour and other reactions

1. Fuse 30 mg of the test substance with 25 mg of potassium nitrate R and 0.05 g of potassium hydroxide R. Cool, dissolve the residue in 10 ml of water and filter. To 5 ml of the filtrate add 1.0 ml of hydrochloric acid (~70 g/l)TS and 0.5 ml of barium chloride (50 g/l)TS; a white precipitate is produced.
2. Dissolve 20 mg of the test substance in 5 ml of water and add 1.5 ml of sodium hydroxide (~80 g/l)TS. Heat until a clear solution is obtained; a characteristic pungent odour is perceptible. Cool the solution and divide it into two portions:
 - a) To 1 portion add 2-3 drops of potassium permanganate (10 g/l)TS; the purple colour changes to violet then to blue and finally to emerald green.
 - b) Acidify the second portion with a few drops of sulfuric acid (~100 g/l)TS, add 2-3 drops of potassium permanganate (10 g/l)TS and shake; the purple colour of potassium permanganate is not discharged.

CARBAMAZEPINE TABLETS

Description. Each tablet usually contains 200 mg of carbamazepine.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.20 g of carbamazepine.
2. Grind the tablets, weigh out the above calculated equivalent amount to carbamazepine as powdered material, shake it with 10 ml of warm acetone R, filter while still warm, evaporate the filtrate to dryness on a water-bath and dry at 80 °C. Use the residue as the test substance.

IDENTITY TESTS

Melting point. The test substance melts at about 189 °C.

Colour and other reactions

1. To 5 mg of the test substance add 1 ml of formaldehyde/sulfuric acid TS; a yellow colour is gradually produced which turns orange on standing.
2. To 10 mg of the test substance add about 2 ml of nitric acid (~1000 g/l)TS and heat in a water-bath for 1 minute; an orange colour is produced.

CHLORAMBUCIL TABLETS

Description. Each tablet usually contains 2-5 mg of chlorambucil. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amount equivalent to 0.05 g of chlorambucil.
2. Grind the tablets or cores, weigh out the above calculated equivalent amount to chlorambucil as powdered material, shake it with 20 ml of chloroform R, filter, and evaporate the filtrate to dryness on a water-bath. Use the residue as the test substance.

IDENTITY TESTS

Colour and other reactions

1. Dissolve 10 ml of the test substance in a mixture of 1.0 ml of acetone R and 1.0 ml of water. Add 1 drop of sulfuric acid (~1760 g/l)TS and a few drops of silver nitrate (40 g/l)TS; no opalescence is immediately observed. Warm the solution on a water-bath for 2-3 minutes; an opalescence is obtained.
2. To 30 mg of the test substance add 3.0 ml of hydrochloric acid (~70 g/l)TS, mix and allow to stand for 30 minutes, shaking occasionally. Filter, wash the residue with 5 ml of water (keep the filtrate for test 3) and dry the residue at 105 °C for 3 hours; melting point, about 146 °C.
3. To 5 ml of the filtrate from test 2 add 0.5 ml of potassio-mercuric iodide TS; a light beige coloured precipitate is produced.

CHLORAMPHENICOL CAPSULES

Description. Each capsule usually contains 250 mg of chloramphenicol.

Preparation of the sample

1. Weigh the contents of 1 capsule and calculate the amount equivalent to 45 mg of chloramphenicol.
2. Empty the capsules, weigh out the above calculated equivalent amount to chloramphenicol and use it directly as the test substance. Divide the test substance into three equal parts.

IDENTITY TESTS

Colour and other reactions

1. To 2 parts of the test substance add 10 ml of water, 2.0 ml of sulfuric acid (~100 g/l)TS and 0.2 g of zinc R powder. Allow to stand for 10 minutes and filter. To 5 ml of the filtrate (keep the remaining filtrate for test 2) add a few drops of nitric acid (~130 g/l)TS and a few drops of silver nitrate (40 g/l)TS; a white, curdy precipitate is produced.
2. To the filtrate from test 1 add 1-2 drops of sodium nitrite (10 g/l)TS, after a few minutes add 1.0 g of urea R and a solution of 10 mg of 2-naphthol R dissolved in 2.0 ml of sodium hydroxide (~80 g/l)TS; a red colour is produced.
3. Add the remaining part of the test substance to a mixture of 5 drops of liquefied phenol R and 0.20 g of potassium hydroxide R. Heat to boiling over a small flame and shake; a brown-red colour is produced. Add 2.0 ml of water; the colour turns to dark green.

CHLORAMPHENICOL PALMITATE ORAL SUSPENSION

Description. The suspension usually contains 2.6 g of chloramphenicol palmitate equivalent to about 1.5 g of chloramphenicol in 50 ml of a suitable vehicle.

Preparation of the sample

Pool the contents of the containers equivalent to about 2 g of chloramphenicol and use it directly as the test solution. Divide the test solution into four equal volumes.

IDENTITY TESTS

Colour and other reactions

1. To 1 volume of the test solution add 10 ml of ethanol (~750 g/l)TS, shake to dissolve and if necessary, heat gently. Add 0.2 g of zinc R powder, 2.0 ml of hydrochloric acid (~250 g/l)TS and allow to stand for 10 minutes. Filter, to the filtrate add 0.5 ml of sodium nitrite (10 g/l)TS and allow to stand for 2 minutes. Following this add 1.0 g of urea R and a solution containing 10 mg of 2-naphthol R in 2.0 ml of sodium hydroxide (~80 g/l)TS; a brownish orange colour is produced.
2. Repeat test 1 but omitting the zinc R powder; no red colour is produced.
3. Dilute 1 volume of the test solution with 10 ml of ethanol (~750 g/l)TS, if necessary, heat slightly, add 0.2 g of zinc R powder, 1.0 ml of sulfuric acid (~100 g/l)TS and allow to stand for 10 minutes. Filter, to the filtrate add a few drops of nitric acid (~130 g/l)TS and a few drops of silver nitrate (40 g/l)TS; a white, curdy precipitate is produced.
4. Boil 1 drop of the test solution with 5 ml of sodium hydroxide (~80 g/l)TS; the mixture foams strongly and a dark yellow colour is produced.

CHLOROQUINE PHOSPHATE TABLETS

Description. Each tablet contains chloroquine phosphate usually equivalent to 150-200 mg of chloroquine. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amounts equivalent to 0.25 g, 0.05 g and 5 mg of chloroquine phosphate.
2. Grind the tablets or cores, weigh out the above calculated equivalent amounts to chloroquine phosphate as powdered material and use them directly: 5 mg for test substance 1; 0.05 g for test substance 2; 0.25 g for test substance 3.
3. Shake test substance 1 with 1.0 ml of water, filter and use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To the test solution add 1.0 ml of silver nitrate (40 g/l)TS; a yellow precipitate is produced. Divide this solution with the precipitate in two parts. To one part add a few drops of nitric acid (~130 g/l)TS; the precipitate dissolves to a clear solution. To the other part add a few drops of ammonia (~100 g/l)TS; the yellow precipitate dissolves but a small amount of white precipitate is produced.
2. Transfer test substance 2 to a small test-tube and fuse it with 1 pellet of potassium hydroxide R for approximately 5 minutes. Drop the red-glowing tube into a conical flask containing 2.0 ml of water. Filter the mixture into another test-tube, acidify with 1.0 ml of nitric acid (~130 g/l)TS and add 5 drops of silver nitrate (40 g/l)TS; an off-white, curdy precipitate is produced.
3. Shake test substance 3 with 20 ml of water, filter and to the filtrate add 5 ml of a saturated solution of trinitrophenol R in water; a yellow precipitate is produced. Filter and wash the precipitate until the washing liquid becomes colourless. Collect the precipitate and dry in a desiccator over sulfuric acid at room temperature; melting behaviour, about 207 °C.

CHLOROQUINE SULFATE TABLETS

Description. Each tablet usually contains 150-200 mg of chloroquine sulfate.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 0.05 g, 20 mg and 15 mg of chloroquine sulfate.
2. Grind the tablets, weigh out the above calculated equivalent amounts to chloroquine sulfate as powdered material and use them directly: 0.05 g for test substance 1; 20 mg for test substance 2; 15 mg for test substance 3.

IDENTITY TESTS

Colour and other reactions

1. Add test substance 1 to 5 ml of sulfuric acid (~1760 g/l)TS; the colour of the mixture remains unchanged. Add 3 drops of potassium dichromate (100 g/l)TS; a red colour is produced which quickly changes to red-brown.
2. Suspend test substance 2 in 2.0 ml of water and add 5 drops of potassio-mercuric iodide TS; a faintly yellow, thick precipitate is produced.
3. Suspend test substance 3 in 5 ml of water, filter, acidify the filtrate with 1.0 ml of hydrochloric acid (~70 g/l)TS and add 1.0 ml of barium chloride (50 g/l)TS; a white precipitate is produced.

CHLORPHENAMINE HYDROGEN MALEATE TABLETS

Description. Each tablet usually contains 4 mg of chlorphenamine hydrogen maleate.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 5 mg, 10 mg and 40 mg of chlorphenamine hydrogen maleate.
2. Grind the tablets, weigh out the above calculated equivalent amounts to chlorphenamine hydrogen maleate as powdered material and use them directly: 10 mg for test substance 1; 2 portions of 5 mg for test substance 2; 40 mg for test substance 3.
3. Shake test substance 1 with 3 ml of sodium nitrite (10 g/l)TS, filter and use the filtrate as test solution 1. Shake test substance 2 with 2.0 ml of water, filter and use the filtrate as test solution 2.

IDENTITY TESTS

Colour and other reactions

1. Shake test substance 2 with 5 ml of chloroform R, filter and evaporate the filtrate on a water-bath. Dissolve the residue in 2.0 ml of water, add 3 drops of ferric chloride (25 g/l)TS and heat to boiling; a yellow-orange colour is produced.
2. Divide test solution 1 equally in two test-tubes. To one tube add 1.0 ml of sulfanilic acid TS. Heat both tubes to boiling; a yellow colour is produced in the treated test solution compared to the remaining very faint pale yellow test solution.
3. To test solution 2 add 1 drop of potassium permanganate (10 g/l)TS; the purple colour is discharged.
4. Add about 20 ml of water to test substance 3, warm it to dissolve and filter. To the filtrate add 10 ml of a saturated solution of trinitrophenol R in water and warm on a water-bath for 5 minutes; a precipitate is produced. Filter, wash the precipitate with water, collect the precipitate and dry it at 105 °C for 1 hour; melting behaviour, about 196 °C with decomposition.

CHLORPROMAZINE HYDROCHLORIDE TABLETS

Description. Each tablet usually contains 100 mg of chlorpromazine hydrochloride. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amounts equivalent to 0.05 g and 10 mg of chlorpromazine hydrochloride.
2. Grind the tablets or cores, weigh out the above calculated equivalent amounts to chlorpromazine hydrochloride as powdered material and use them directly: 0.05 g for test substance 1; 10 mg for test substance 2.
3. Shake test substance 1 with 5 ml of water, filter and use the filtrate as test solution 1. Shake test substance 2 with 5 ml of chloroform R, filter and use the filtrate as test solution 2.

IDENTITY TESTS

Colour and other reactions

1. To 0.5 ml of test solution 1 add carefully 2 ml of sulfuric acid (~1760 g/l)TS and allow to stand for 5 minutes; a red colour is produced (distinction from promazine).
2. To test solution 2 add 1.0 ml of sodium metaperiodate (60 g/l)TS and 2.0 ml of sulfuric acid (~100 g/l)TS. Shake vigorously and allow the layers to separate; a vivid red colour is produced in the aqueous layer that fades slowly on standing and the chloroform layer acquires a pink colour (distinction from promethazine).
3. To 1.0 ml of test solution 1 add 4 ml of water, about 1.5 ml of sodium hydroxide (~80 g/l)TS, mix and filter. To the filtrate add 1.0 ml of nitric acid (~130 g/l)TS and a few drops of silver nitrate (40 g/l)TS; a white precipitate is produced. Add 1.0 ml of ammonia (~100 g/l)TS; the precipitate dissolves.

CLOMIFENE CITRATE TABLETS

Description. Each tablet usually contains 50 mg of clomifene citrate.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 10 mg of clomifene citrate.
2. Grind the tablets, weigh out the above calculated equivalent amount to clomifene citrate as powdered material and use it directly as the test substance. Divide the test substance into four equal parts.

IDENTITY TESTS

Colour and other reactions

1. To 1 part of the test substance add 1 drop of sulfuric acid (~1760 g/l)TS; an orange colour is first produced which slowly changes to yellowish brown and finally to greenish brown.
2. To 1 part of the test substance add 1 ml of formaldehyde/sulfuric acid TS; a purplish brown colour is produced.
3. Dissolve 2 parts of the test substance in 5 ml of a mixture of 1 volume of acetic anhydride R and 5 volumes of pyridine R, and heat in a water-bath; a deep wine red colour is produced.

CODEINE PHOSPHATE TABLETS

Description. Each tablet usually contains 10-30 mg of codeine phosphate.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 60 mg of codeine phosphate.
2. Grind the tablets, weigh out the above calculated equivalent amount to codeine phosphate as powdered material, shake it with 4 portions of 10 ml of ethanol (~750 g/l)TS and filter. Evaporate the combined filtrate to dryness on a water-bath and use the residue as the test substance.

IDENTITY TESTS

Colour and other reactions

1. Dissolve 10 mg of the test substance in 5 ml of sulfuric acid (~1760 g/l)TS, add 1 drop of ferric chloride (25 g/l)TS and if necessary, heat gently; a violet-blue colour is produced. Add a few drops of nitric acid (~130 g/l)TS; the colour changes to dark red.
2. Dissolve about 1 mg of the test substance in 1 ml of formaldehyde/sulfuric acid TS; a dark violet colour is immediately produced.
3. Dissolve 20 mg of the test substance in 1.0 ml of water and add 1 drop of ferric chloride (25 g/l)TS; a precipitate is formed but no blue tinge is observed in the solution (distinction from morphine).
4. Dissolve 10 mg of the test substance in 2.0 ml of freshly boiled and cooled water and add a few drops of silver nitrate (40 g/l)TS; a yellow precipitate is produced. Divide the solution with the precipitate into two portions. To one portion add a few drops of nitric acid (~130 g/l)TS; the precipitate dissolves to a clear solution. To the other portion add a few drops of ammonia (~100 g/l)TS and shake well; the precipitate dissolves as well to a clear solution.

COLCHICINE TABLETS

Description. Each tablet usually contains 0.5 mg of colchicine.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 3 mg of colchicine.
2. Grind the tablets, weigh out the above calculated equivalent amount to colchicine as powdered material and use it directly as the test substance. Divide the test substance into three equal parts.

IDENTITY TESTS

Colour and other reactions

1. Suspend 1 part of the test substance in 2.0 ml of water and filter; the filtrate is colourless. Add a few drops of hydrochloric acid (~70 g/l)TS; the colour of the solution turns to yellow.
2. To 1 part of the test substance add 1.0 ml of hydrochloric acid (~70 g/l)TS, heat to boiling for 2 minutes and add 3 drops of ferric chloride (25 g/l)TS; a deep yellowish green colour is produced.
3. Suspend the remaining part of the test substance in 1.5 ml of ethanol (~750 g/l)TS and filter. Place a few drops of the filtrate onto a porcelain dish and evaporate to dryness on a water-bath. Mix the residue with 3 drops of sulfuric acid (~1760 g/l)TS; a lemon yellow colour is produced. Add 1 drop of nitric acid (~1000 g/l)TS; the colour changes to greenish blue, turning rapidly to reddish and finally to yellowish. Following this add about 0.5 ml of sodium hydroxide (~200 g/l)TS; the colour turns to red.

CYCLOPHOSPHAMIDE TABLETS

Description. Each tablet usually contains 25 mg of cyclophosphamide. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amounts equivalent to 0.20 g and 0.10 g of cyclophosphamide.
2. Grind the tablets or cores, weigh out the above calculated equivalent amounts to cyclophosphamide as powdered material and use them directly: 0.20 g for test substance 1; 0.10 g for test substance 2. Shake test substance 1 with 20 ml of ethanol (~750g/l)TS, filter, evaporate the filtrate to dryness on a water-bath and use the residue as the test substance.
3. Shake test substance 2 with 10 ml of water, filter and use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To 5 ml of the test solution add 5 ml of silver nitrate (40 g/l)TS; not more than a slightly opalescent solution is produced. Boil; a white precipitate is formed which is insoluble in nitric acid (~130 g/l)TS, but is soluble in ammonia (~100 g/l)TS.
2. Fuse the test substance with 0.06 g of potassium nitrate R and 0.08 g of potassium hydroxide R. Dissolve the residue in 20 ml of water and filter. To 5 ml of the filtrate add 1.5 ml of hydrochloric acid (~70 g/l)TS and 1.0 ml of ammonium molybdate (95 g/l)TS; a yellow precipitate is produced.

CYTARABINE INJECTION

Description. The injection is a sterile solution usually containing 20 mg of cytarabine in 1.0 ml of a suitable vehicle.

Preparation of the sample

Pool the contents of the ampoules equivalent to 0.10 g of cytarabine and use it directly as the test solution. Divide the test solution into five equal volumes.

IDENTITY TESTS

Colour and other reactions

1. To 2 volumes of the test solution add 0.5 ml of hydrochloric acid (~70 g/l)TS and 4-5 drops of sodium nitrite (10 g/l)TS, and shake well. After 2-3 minutes add 4-5 drops of 2-naphthol TS; a reddish yellow precipitate is produced. Shake the mixture; the colour of the precipitate turns to greenish black while the solution becomes reddish.
2. Dilute 1 volume of the test solution with 1.0 ml of water, add 0.5 ml of sodium hydroxide (~80 g/l)TS and heat in a water-bath for 3 minutes. Cool and add 0.5 ml of copper(II) acetate (45 g/l)TS; a green precipitate is formed. Warm on a water-bath for 5 minutes; the colour of the precipitate turns reddish.
3. Dilute 2 volumes of the test solution with 5 ml of water and add 1.0 ml of hydrochloric acid (~70 g/l)TS. Warm on a water-bath for 5 minutes, cool and transfer into each of 2 test-tubes half of the solution:
 - a) To one test-tube add a few drops of potassium permanganate (10 g/l)TS; the purple colour is discharged.
 - b) To the other test-tube add 0.5 ml of bromine TS and shake; the colour of bromine is discharged.

DAPSONE TABLETS

Description. Each tablet usually contains 50-100 mg of dapsone.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.10 g of dapsone.
2. Grind the tablets, weigh out the above calculated equivalent amount to dapsone as powdered material, shake it with 10 ml of warm acetone R, filter, evaporate the filtrate, dry at 105 °C for 30 minutes and use the residue as the test substance.

IDENTITY TESTS

Melting point. The test substance melts at about 174 °C.

Colour and other reactions

1. Dissolve 0.05 g of the test substance in 2.0 ml of hydrochloric acid (~70 g/l)TS, cool in ice and add 4 ml of sodium nitrite (10 g/l)TS. Allow to stand for 2 minutes then pour the mixture into 2.0 ml of freshly prepared 2-naphthol TS containing 1.0 g of sodium acetate R; an orange-red precipitate is produced.
2. Dissolve 10 mg of the test substance in 1.0 ml of hydrochloric acid (~70 g/l)TS, add 5 ml of water, about 0.2 ml of formaldehyde TS and mix; a milky suspension is produced which changes rapidly into a white precipitate.

DEXAMETHASONE TABLETS

Description. Each tablet usually contains 0.5-4.0 mg of dexamethasone.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to about 1 mg and 5 mg of dexamethasone.
2. Grind the tablets, weigh out the above calculated equivalent amounts to dexamethasone as powdered material and use them directly: 2 portions of about 1 mg for test substance 1; 5 mg for the preparation of test substance 2, incinerate the material with 1.0 g of calcium oxide R and use the white ashes obtained.

IDENTITY TESTS

Colour and other reactions

1. To 1 portion of test substance 1 add 2.0 ml of ethanol (~750 g/l)TS and shake. Then add 1.0 ml of potassio-cupric tartrate TS and heat to boiling; an orange precipitate is slowly produced.
2. Mix 2 drops of potassium dichromate (100 g/l)TS with 3 ml of sulfuric acid (~1760 g/l)TS and heat on a water-bath for 5 minutes; the solution wets the sides of the tube. Add test substance 2 to this solution, shake well and heat again for 5 minutes on a water-bath; the colour turns violet-black and the solution no longer wets the sides of the tube.
3. To 1 ml of formaldehyde/sulfuric acid TS add the remaining portion of test substance 1 and shake well; a yellow-orange colour is produced. Heat on a water-bath for 1 minute; the colour changes to dark brown.

DIAZEPAM TABLETS

Description. Each tablet usually contains 5 mg of diazepam. The tablets may contain a dye.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.20 g of diazepam.
2. Grind the tablets, weigh out the above calculated equivalent amount to diazepam as powdered material, shake it with 2 volumes of chloroform R, each of 5 ml, filter, evaporate the combined filtrate to dryness on a water-bath and use the residue as the test substance.

IDENTITY TESTS

Colour and other reactions

1. Dissolve 0.10 g of the test substance in 3 ml of hydrochloric acid (~250 g/l)TS and heat on a water-bath for 30 minutes; a brownish yellow solution is produced. Cool, dilute with about 10 ml of ice-water, allow to precipitate placing it in a refrigerator overnight, filter the crystalline precipitate, wash with water, and dry at 80 °C for 2 hours; melting point about 94 °C.
2. To 0.05 g of the test substance add 10 mg of triketohydrindene hydrate R and 0.5 ml of ethanol (~750 g/l)TS; a pale yellowish green colour is produced. Following this add 2 drops of a mixture composed of 2 drops of copper(II) sulfate (160 g/l)TS and 3 ml of water; the colour changes to yellow-orange.
3. To about 10 mg of the test substance add 5 ml of sulfuric acid (~5 g/l)TS and heat on a water-bath for 3-5 minutes; a pale yellow colour is produced.

DIETHYLCARBAMAZINE DIHYDROGEN CITRATE TABLETS

Description. Each tablet usually contains 50 mg of diethylcarbamazine dihydrogen citrate.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.5 g of diethylcarbamazine dihydrogen citrate.
2. Grind the tablets, weigh out the above calculated equivalent amount to diethylcarbamazine dihydrogen citrate as powdered material and use it directly as the test substance.

IDENTITY TESTS

Colour and other reactions

1. Extract thoroughly the test substance with two successive portions of 10 ml of water, filter, transfer the combined filtrate to a separator, make the

solution alkaline with sodium hydroxide (~200 g/l)TS and extract with 4 successive quantities, each of 5 ml of chloroform R (retain the aqueous layer for test 2). Wash the combined chloroform extracts with water, filter through a plug of cotton wool and evaporate to dryness on a water-bath. To the residue add 1.0 ml of ethyl iodide R, heat gently under a reflux condenser for 5 minutes, cool and separate the viscous yellow oil. Dissolve it in a small volume of ethanol (~750 g/l)TS, chill the solution in an ice-bath and while stirring add sufficient ethyl acetate R to obtain a precipitate. Stir until it crystallizes and allow the mixture to remain in the ice-bath for 30 minutes. Filter and dry the precipitate at 105 °C; melting point about 152 °C.

2. Neutralize the aqueous layer retained in test 1 with sulfuric acid (~100 g/l)TS, add an excess of mercuric sulfate TS and boil. Add a few drops of potassium permanganate (10 g/l)TS; the initial colour is discharged and a white precipitate is produced.

DIGOXIN TABLETS

Description. Each tablet usually contains 0.0625-0.25 mg of digoxin.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to about 1 mg of digoxin.
2. Grind the tablets, weigh out the above calculated equivalent amount to digoxin as powdered material and use it directly as the test substance. Divide the test substance into two equal parts.

IDENTITY TESTS

Colour and other reactions

1. To 1 part of the test substance add 2.0 ml of a solution prepared by mixing 0.5 ml of ferric chloride (25 g/l)TS with 100 ml of glacial acetic acid, R and shake. Filter and superimpose the filtrate onto 1 ml of sulfuric acid (~1760 g/l)TS; a brown ring, but no red colour is produced at the junction of the two liquids and after some time the acetic acid layer acquires a bluish green colour.
2. To the remaining part of the test substance add 5 ml of ethanol (~750 g/l)TS and 3 ml of alkaline trinitrophenol TS; a yellow colour is produced, which darkens with time.

EPHEDRINE HYDROCHLORIDE TABLETS

Description. Each tablet usually contains 30 mg of ephedrine hydrochloride.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.20 g of ephedrine hydrochloride.

2. Grind the tablets, weigh out the above calculated equivalent amount to ephedrine hydrochloride as powdered material, shake it with 10 ml of water, filter, evaporate the filtrate almost to dryness, allow to crystallize from water, separate the crystals, dry them at 105 °C for 1 hour and use the dried material as the test substance. Divide the test substance into four equal parts.

IDENTITY TESTS

Melting point. The test substance melts at about 186 °C.

Colour and other reactions

1. Dissolve 1 part of the test substance in 1.0 ml of water, add 1-2 drops of copper(II) sulfate (160 g/l)TS and 1.0 ml of sodium hydroxide (~80 g/l)TS; a violet colour is produced. To this solution add 2.0 ml of 1-butanol R and shake; the butanol layer acquires a reddish violet colour.
2. Dissolve 1 part of the test substance in 5 ml of water, add a few drops of sodium hydroxide (~80 g/l)TS, 3 ml of potassium ferricyanide (50 g/l)TS and heat on a water-bath; a characteristic odour of benzaldehyde is perceptible.
3. Dissolve 1 part of the test substance in 2.0 ml of water, add a few drops of nitric acid (~130 g/l)TS and a few drops of silver nitrate (40 g/l)TS; a white, curdy precipitate is produced.

EPINEPHRINE HYDROCHLORIDE OPHTHALMIC SOLUTION

Description. The solution usually contains 2.0 mg of epinephrine hydrochloride in 1.0 ml of a suitable vehicle.

Preparation of the sample

Pool the contents of the containers equivalent to 40 mg of epinephrine hydrochloride and use it directly as the test solution. Divide the test solution into four equal volumes.

IDENTITY TESTS

Colour and other reactions

1. To 1 volume of the test solution add 1-2 drops of ferric chloride (25 g/l)TS; a green or emerald green colour is produced. Add 1 drop of ammonia (~260 g/l)TS; the colour of the solution changes to red.
2. To 1 volume of the test solution add 2.0 ml of water and 0.5 ml of sodium nitrite (10 g/l)TS; within 10 minutes a deep red colour is produced.
3. Add 2.0 ml of nitric acid (~130 g/l)TS to 1 volume of the test solution and shake well. Add 0.5 ml of silver nitrate (40 g/l)TS; a white precipitate is produced.
4. To 1 volume of the test solution add 2-3 drops of sulfuric acid (~1760 g/l)TS, 2.0 ml of ammonium molybdate (95 g/l)TS and mix; a yellow colour develops. Add 2.0 ml of sodium hydroxide (~80 g/l)TS; the colour of the solution changes to greenish yellow.

ERGOTAMINE TARTRATE TABLETS

Description. Each tablet usually contains 2.0 mg of ergotamine tartrate. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amount equivalent to 2.0 mg of ergotamine tartrate.
2. Grind the tablets or cores, weigh out the above calculated equivalent amount to ergotamine tartrate as powdered material, shake it with 20 ml of tartaric acid (10 g/l)TS, filter and use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. The test solution shows a blue fluorescence in ultraviolet light.
2. To 1.0 ml of the test solution add slowly 2.0 ml of 4-dimethylaminobenzaldehyde TS and mix; a blue colour is slowly produced.
3. To 2.0 ml of the test solution add 2 drops of potassio-mercuric iodide TS; a white turbidity is produced (distinction from ergometrine maleate).

ETHAMBUTOL HYDROCHLORIDE TABLETS

Description. Each tablet usually contains 100-500 mg of ethambutol hydrochloride.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 0.075 g and 20 mg of ethambutol hydrochloride.
2. Grind the tablets, weigh out the above calculated equivalent amounts to ethambutol hydrochloride as powdered material and use them directly: 0.075 g for test substance 1; 20 mg for test substance 2.

IDENTITY TESTS

Colour and other reactions

1. Shake test substance 1 with 5 ml of water and filter. To the filtrate add 2-4 drops of copper(II) sulfate (160 g/l)TS and 1.0 ml of sodium hydroxide (~80 g/l)TS; a distinct blue solution is produced.
2. Suspend test substance 2 in 5 ml of water and filter. To the filtrate add 0.5 ml of nitric acid (~130 g/l)TS and 1.0 ml of silver nitrate (40 g/l)TS; a white precipitate is produced. Add a few drops of ammonia (~100 g/l)TS; the precipitate dissolves.

ETHINYLESTRADIOL TABLETS

Description. Each tablet usually contains 0.05 mg of ethinylestradiol. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amount equivalent to 2.5 mg of ethinylestradiol.
2. Grind the tablets or cores, weigh out the above calculated equivalent amount to ethinylestradiol as powdered material, shake it with 25 ml of dehydrated ethanol R, and filter. Use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. Evaporate 10 ml of the test solution to dryness on a water-bath. To the residue add about 1 ml of sulfuric acid (~1760 g/l)TS; an orange solution is produced. Dilute the solution with 10 ml of water; a red-violet colour is produced.
2. Evaporate 10 ml of the test solution to dryness on water-bath. To the residue add 0.5 ml of ethanol (~750 g/l)TS and about 1 ml of sulfuric acid (~1760 g/l)TS; an orange solution is produced which shows a green fluorescence. Dilute the solution with 5 ml of ethanol (~750 g/l)TS; a red-violet solution is produced which shows a green fluorescence.

FERROUS SULFATE TABLETS

Description. Each tablet contains ferrous sulfate or dried ferrous sulfate usually equivalent to 60 mg of iron. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amount equivalent to 0.3 g of ferrous sulfate.
2. Grind the tablets or cores, weigh out the above calculated equivalent amount to ferrous sulfate as powdered material and use it directly as the test substance. Divide the test substance into three equal parts.

IDENTITY TESTS

Colour and other reactions

1. To 1 part of the test substance add 5 ml of water, then drop by drop ammonia (~100 g/l)TS until a bluish-green precipitate is produced. Shake the mixture vigorously; the precipitate turns dark green, then greenish-brown and on standing brown.

2. Add 1 part of the test substance to a mixture composed of 3 ml of water and 4 drops of hydrochloric acid (~70 g/l)TS, then add 1.0 ml of potassium ferricyanide (50 g/l)TS; a dark blue precipitate is produced.

Alternate colour test:

Sprinkle a few crystals of potassium ferricyanide R on a small quantity of moistened test substance; deep blue spots appear.

3. Add the remaining test substance to a mixture composed of 3 ml of water and 4 drops of hydrochloric acid (~70 g/l)TS, shake well and filter. Then add to the filtrate 1.0 ml of barium chloride (50 g/l)TS; a white precipitate is produced.

FOLIC ACID TABLETS

Description. Each tablet usually contains 1.0 mg of folic acid.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 10 mg and 40 mg of folic acid.
2. Grind the tablets, weigh out the above calculated equivalent amounts to folic acid as powdered material and use them directly: 10 mg for test substance 1; 40 mg for test substance 2.

IDENTITY TESTS

Colour and other reactions

1. To test substance 1 add 2.0 ml of hydrochloric acid (~70 g/l)TS, shake and filter. To the filtrate add 2 drops of liquified phenol R and 3 drops of potassium bromate (15 g/l)TS; a dark red colour is produced.
2. Prepare a suspension by adding 18 ml of water and 2.0 ml of ammonia (~100 g/l)TS to test substance 2 and shake. To test-tube 1 transfer 5 ml of the suspension and add 0.5 ml of potassium permanganate (10 g/l)TS. To test-tube 2 transfer 1.0 ml of the suspension and 5 ml of water. To both tubes add 2.0 ml of sodium nitrite (10 g/l)TS and 2.5 ml of hydrochloric acid (~70 g/l)TS, mix and allow to stand for 2 minutes. Then add to both tubes 0.5 ml of a solution prepared by dissolving 0.25 g of 2-naphthol R in 15 ml of sodium hydroxide (~80 g/l)TS; an intense reddish brown colour is produced in test-tube 1, whereas a yellowish colour is formed in test-tube 2.

FUROSEMIDE INJECTION

Description. The injection is a sterile solution usually containing 10 mg of furosemide in 1.0 ml of a suitable vehicle.

Preparation of the sample

Pool the contents of the ampoules equivalent to 40 mg of furosemide, evaporate to dryness on a water-bath, shake the residue with 10 ml of acetone R and filter. Evaporate the filtrate to dryness on a water-bath and use the residue as the test substance.

IDENTITY TESTS

Colour and other reactions

1. To 25 mg of the test substance add 5 ml of ethanol (~750 g/l)TS, heat on a water-bath for 2-3 minutes and add 3.0 ml of 4-dimethylaminobenzaldehyde TS without shaking; a yellow-green colour is produced which changes to red starting from the bottom of the test-tube.
2. Dissolve 5 mg of paraformaldehyde R in about 1 ml of sulfuric acid (~1760 g/l)TS and add to it 5 mg of the test substance; a yellow colour is obtained (retain the solution for test 3).
3. Heat the solution from test 2 on a water-bath for 5 minutes; the colour of the solution changes to red-brown. Carefully add 10 ml of water; the colour changes to light green.

FUROSEMIDE TABLETS

Description. Each tablet usually contains 40 mg of furosemide.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 40 mg and 5 mg of furosemide.
2. Grind the tablets, weigh out the above calculated equivalent amounts to furosemide as powdered material and use them directly: 40 mg for test substance 1; 5 mg for test substance 2.
3. Shake test substance 1 with 10 ml of ethanol (~750 g/l)TS, filter and use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To 5 ml of the test solution add 2.0 ml of 4-dimethylaminobenzaldehyde TS; a deep red colour is produced.
2. Dissolve 5 mg of paraformaldehyde R in 1 ml of sulfuric acid (~1760 g/l)TS and add to it test substance 2; a deep yellow colour is produced with a brownish tinge (retain the solution for test 3).
3. Heat the solution obtained in test 2 on a water-bath for 5 minutes; the colour of the solution changes to red-brown.

GLUCOSE INJECTION

Description. The injection is a sterile solution usually containing 50-500 mg of glucose in 1.0 ml of a suitable vehicle.

Preparation of the sample

1. Pool the contents of the containers equivalent to 0.25 g of glucose and use it directly as the test solution.
2. Evaporate the equivalent amount of about 0.25 g of glucose on a water-bath and use the viscous residue as the test substance.

IDENTITY TESTS

Heating behaviour. Heat gently a small quantity of the test substance; it becomes yellow, then brown, and an odour of burnt sugar is perceptible. Heat further to ignition; the melt swells, then burns and chars.

Colour and other reactions

To 5 ml of the test solution add 1.0 ml of potassium cupric tartrate TS and heat on a water-bath for 10 minutes; a brick red precipitate is formed.

GRISEOFULVIN TABLETS

Description. Each tablet usually contains 125-250 mg of griseofulvin.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 20 mg of griseofulvin.
2. Grind the tablets, weigh out the above calculated equivalent amount to griseofulvin as powdered material and use it directly as the test substance. Divide the test substance into four equal parts.

IDENTITY TESTS

Melting behaviour. The test substance melts at about 218 °C with decomposition.

Colour and other reactions

1. Dissolve 2 parts of the test substance in 1.0 ml of ethanol (~750 g/l)TS; add 0.20 g of sodium sulfite R, 1.0 ml of sodium hydroxide (~80 g/l)TS, heat on a water-bath and allow to stand for about 10 minutes; a lemon-yellow colour is produced.
2. Dissolve 1 part of the test substance in about 1 ml of sulfuric acid (~1760 g/l)TS; a yellow-orange colour is produced. Add 1 drop of potassium dichromate (1000 g/l)TS; the colour of the solution changes to wine-red.

HOMATROPINE HYDROBROMIDE OPHTHALMIC SOLUTION

Description. The solution usually contains 20 mg of homatropine hydrobromide in 1.0 ml of a suitable vehicle.

Preparation of sample

Pool the contents of the containers equivalent to 50 mg of homatropine hydrobromide, evaporate it to dryness on a water-bath and use the residue as the test substance.

IDENTITY TESTS

Colour and other reactions

1. Heat cautiously in a dry test-tube 10 mg of the test substance with 2-3 drops of sulfuric acid (~1760 g/l)TS until a brown colour is produced. Cool, add 5 ml of water and boil; an odour of benzaldehyde is perceptible.
2. Place 5 mg of the test substance in a porcelain dish, add about 0.5 ml of nitric acid (~1000 g/l)TS and about 0.5 ml of acetic anhydride R, and evaporate to dryness on a water-bath; a yellow residue is obtained. To the cooled residue add 0.5 ml of acetone R and a few drops of potassium hydroxide/ethanol TS; a violet colour is produced which disappears on standing.
3. Repeat test 2 without the addition of acetic anhydride R; no violet colour is produced (distinction from atropine and hyoscyne).

IBUPROFEN TABLETS

Description. Each tablet usually contains 200 mg of ibuprofen.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.5 g of ibuprofen.
2. Grind the tablets, weigh out the above calculated equivalent amount to ibuprofen as powdered material, shake it with 20 ml of acetone R, filter, allow the filtrate to evaporate to dryness without heating, crystallize the residue from 10 ml of acetone R, separate the crystals, allow to dry in air and use the residue as the test substance.

IDENTITY TESTS

Melting point. The test substance melts at about 76 °C.

Colour and other reactions

1. Dissolve 30 mg of the test substance in 2.0 ml of ethanol (~750 g/l)TS, dilute with 2.0 ml of water and add 0.05 g of sodium hydrogen carbonate R; a gas is evolved.
2. To 0.06 g of the test substance add 6 drops of thionyl chloride R and heat on a water-bath for 30 minutes. Separately dissolve 0.30 g of hydroxylamine hydrochloride R and 1 pellet of potassium hydroxide R in 3 ml of methanol R. Filter and transfer 2.0 ml of the filtrate to the above mixture. Heat carefully on a water-bath for 2 minutes, add 1.0 ml of hydrochloric acid (~70 g/l)TS and 3 drops of ferric chloride (25 g/l)TS; a brownish red solution is produced.

INDOMETACIN CAPSULES

Description. Each capsule usually contains 25 mg of indometacin.

Preparation of the sample

1. Weigh the contents of 1 capsule and calculate the amount equivalent to 0.06 g of indometacin.
2. Empty the capsules, weigh out the above calculated equivalent amount to indometacin and use it directly as the test substance. Divide the test substance into three equal parts.

IDENTITY TESTS

Colour and other reactions

1. To 1 part of the test substance add 1.0 ml of water, 1 drop of sodium hydroxide (~80 g/l)TS, shake and filter. To the filtrate add 1.0 ml of sodium nitrite (10 g/l)TS, allow to stand for 5 minutes and cautiously add about 0.5 ml of hydrochloric acid (~250 g/l)TS; a green colour is produced.
2. To the remaining parts of the test substance add 2.0 ml of sodium hydroxide (~80 g/l)TS; a strong yellow colour is produced that changes gradually to colourless.

IODINE SOLUTION

Description. The solution usually contains 20 mg of iodine and 25 mg of potassium iodide or sodium iodide in 1.0 ml of a suitable vehicle.

Preparation of the sample

1. Pool the contents of the containers equivalent to 0.06 g of iodine and use it directly as the test solution.
2. Evaporate half of the volume of the test solution to dryness on a water-bath, ignite gently to volatilize any free iodine and use the residue as the test substance.

IDENTITY TESTS

Colour and other reactions

1. Dilute 1 drop of the test solution with 10 ml of water and add 1.0 ml of starch TS; a deep blue colour is produced.
2. Dissolve 5 mg of the test substance in 2.0 ml of water and add 5 drops of silver nitrate (40 g/l)TS; a yellow, curdy precipitate is formed which is practically insoluble in ammonia (~100 g/l)TS and in nitric acid (~130 g/l)TS.
3. Dissolve 5 mg of the test substance in 2.0 ml of water, acidify with 0.5 ml of hydrochloric acid (~70 g/l)TS, add 5 drops of ferric chloride (25 g/l)TS, 2.0 ml of chloroform R and shake; the chloroform layer acquires a violet colour.

If the solution contains potassium iodide perform the following test:

4. To 5 mg of the test substance add 1.0 ml of water, shake to dissolve and place 1 drop of this solution on a white tile. Sprinkle a few crystals of sodium cobaltinitrite R onto it; a precipitate or turbidity appears (potassium).

If the solution contains sodium iodide perform the following test:

5. Shake 10 mg of the test substance with 1.0 ml of water, acidify with 1-2 drops of acetic acid (~300 g/l)TS and add 2.0 ml of magnesium uranyl acetate TS or zinc uranyl acetate TS; a light yellow, crystalline precipitate is produced (sodium).

ISONIAZID TABLETS

Description. Each tablet usually contains 100-300 mg of isoniazid.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 0.10 g and 40 mg of isoniazid.
2. Grind the tablets, weigh out the above calculated equivalent amounts to isoniazid as powdered material and use them directly: two portions of 0.10 g for test substance 1; 40 mg for test substance 2.

IDENTITY TESTS

Colour and other reactions

1. To one portion of test substance 1 add 2.0 ml of water, shake and filter, then add a mixture composed of 1.0 ml of silver nitrate (40 g/l)TS and 1.0 ml of ammonia (~100 g/l)TS; bubbles of nitrogen evolve, the mixture turns from yellow to black and a metallic silver mirror appears on the sides of the test-tube.
2. To the remaining portion of test substance 1 add 1.0 ml of water, 1.0 ml of sodium hydroxide (~80 g/l)TS and shake; a dense mixture is formed. Following this add a few drops of iodine TS; a transient blue colour may be observed due to the presence of starch and bubbles of gas are evolved.
3. Mix test substance 2 with 0.10 g of anhydrous sodium carbonate R, place it in a dry test-tube and heat; pyridine, perceptible by its odour, is produced (some pharmaceutical aids may mask the odour).

ISOPRENALINE SULFATE TABLETS

Description. Each tablet usually contains 10-20 mg of isoprenaline sulfate.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 25 mg of isoprenaline sulfate.

2. Grind the tablets, weigh out the above calculated equivalent amount to isoprenaline sulfate as powdered material and use it directly as the test substance. Divide the test substance into five equal parts.

IDENTITY TESTS

Colour and other reactions

1. Suspend 2 parts of the test substance in 10 ml of water and add 1-2 drops of ferric chloride (25 g/l)TS; an emerald-green solution is produced. Add a few drops of a freshly prepared solution of 0.10 g of sodium hydrogen carbonate R dissolved in 1.0 ml of water; the colour of the suspension turns slowly to blue and then to red.
2. Suspend 1 part of the test substance in a mixture of 1.0 ml of sulfuric acid (~5 g/l)TS and 2.0 ml of water. Add 3 ml of ammonium molybdate (95 g/l)TS; a yellow colour is produced. While mixing add slowly 2.0 ml of sodium hydroxide (~80 g/l)TS; the yellow colour is discharged, a red colour appears which gradually changes to greenish yellow with a brownish tinge.
3. Suspend 2 parts of the test substance in 5 ml of water, add 1.0 ml of hydrochloric acid (~70 g/l)TS and 1.0 ml of barium chloride (50 g/l)TS; a white, crystalline precipitate is produced.

LEVODOPA TABLETS

Description. Each tablet usually contains 250 mg of levodopa.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 0.15 g and 5 mg of levodopa.
2. Grind the tablets, weigh out the above calculated equivalent amounts to levodopa as powdered material and use them directly: 0.15 g for test substance 1; 5 mg for test substance 2.

IDENTITY TESTS

Colour and other reactions

1. Dissolve test substance 1 in 5 ml of hot water. Divide the solution into two parts. To one part add a few drops of ferric chloride (25 g/l)TS; a dark green solution is produced (retain the remaining part for test 3).
2. Dissolve test substance 2 in 1.0 ml of sulfuric acid (~5 g/l)TS, add 2.0 ml of water and 3 ml of ammonium molybdate (95 g/l)TS and mix; a golden yellow colour appears. Add slowly while mixing 2.0 ml of sodium hydroxide (~80 g/l)TS and allow to stand for about 5 minutes; a yellowish red solution is produced.
3. To the remaining part of the solution obtained in test 1 add about 0.5 mg of triketohydrindene hydrate R and warm on a water-bath; a blue-violet colour is produced.

LIDOCAINE HYDROCHLORIDE INJECTION

Description. The injection is a sterile solution usually containing 10-20 mg of lidocaine hydrochloride in 1.0 ml of a suitable vehicle.

Preparation of the sample

1. Pool the contents of the ampoules equivalent to 0.30 g of lidocaine hydrochloride and use it directly as the test solution. Divide the test solution into four equal volumes.
2. Evaporate 2 volumes of the test solution to dryness on a water-bath and use the residue as the test substance.

IDENTITY TESTS

Colour and other reactions

1. Dissolve the test substance in 10 ml of water. Make the solution just alkaline with sodium hydroxide (~80 g/l)TS using pH-indicator paper R. Collect the precipitate on a small filter, wash it with water and transfer the crystals to a test-tube. Add 1.0 ml of ethanol (~750 g/l)TS, 3-4 drops of cobalt(II) chloride (30 g/l)TS, and shake; a blue precipitate is produced the colour of which gradually changes to green.
2. To 1 volume of the test solution add 3 drops of iodine TS; a brown precipitate is formed.
3. To 1 volume of the test solution add 1.0 ml of nitric acid (~130 g/l)TS and 1.0 ml of silver nitrate (40 g/l)TS; a white precipitate is produced.

MANNITOL INJECTION

Description. The injection is a sterile solution usually containing 100-200 mg of mannitol in 1.0 ml of a suitable vehicle.

Preparation of the sample

Pool the contents of the containers equivalent to 2.0 g of mannitol, evaporate to dryness on a water-bath and use the viscous residue as the test substance. Divide the test substance into four equal parts.

IDENTITY TESTS

Melting point. Dry 1 part of the test substance at 105 °C; it melts at about 167 °C.

Colour and other reactions

1. Add 1 part of the test substance to 2.0 ml of ferric chloride (25 g/l)TS = tube 1. Transfer 5 drops of water to a separate test-tube containing 2.0 ml of ferric chloride (25 g/l)TS = tube 2. To both tubes add 5 drops of sodium hydroxide (~200 g/l)TS; a brown precipitate is formed in tube 2 and a yellow precipitate is formed in tube 1. Shake both tubes vigorously; a clear solution results in tube 1, but the precipitate remains in tube 2. Further addition of sodium hydroxide (~200 g/l)TS does not cause precipitation in tube 1, but more precipitation takes place in tube 2.

2. Dissolve 1 part of the test substance in 2.0 ml of water, add 2.0 ml of potassio-cupric tartrate TS and heat the mixture to boiling; no precipitate is formed.
3. Dissolve the remaining parts of the test substance in 2.0 ml of water, add 1 drop of sodium hydroxide (~80 g/l)TS and 5 drops of potassium permanganate (10 g/l)TS. Heat the mixture to boiling; the solution is decolorized. Following this add 2.0 ml of potassio-cupric tartrate TS and heat again to boiling; a brick red precipitate separates.

MEBENDAZOLE TABLETS

Description. Each tablet usually contains 100 mg of mebendazole.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.08 g of mebendazole.
2. Grind the tablets, weigh out the above calculated equivalent amount to mebendazole as powdered material and use it directly as the test substance. Divide the test substance into two equal parts.

IDENTITY TESTS

Colour and other reactions

1. Shake 1 part of the test substance with 2.0 ml of sodium hydroxide (~80 g/l)TS and heat the yellowish coloured suspension until dissolved; the solution is yellow. Add a few drops of copper(II) sulfate (160 g/l)TS; a greenish precipitate is produced. Add a few drops of ammonia (~100 g/l)TS; the colour of the precipitate turns to greenish blue.
2. To the remaining part of the test substance add 2.0 ml of sulfuric acid (~1760 g/l)TS; a yellow solution is produced. Carefully dilute with 3 ml of water; the yellow colour disappears. Filter and add 1.0 ml of silver nitrate (40 g/l)TS; a white precipitate is formed which does not dissolve in an excess of ammonia (~100 g/l)TS.

METHYLDOPA TABLETS

Description. Each tablet usually contains 250 mg of methyldopa. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amounts equivalent to about 2 mg, 10 mg and 0.04 g of methyldopa.
2. Grind the tablets or cores, weigh out the above calculated equivalent amounts to methyldopa as powdered material and use them directly: about 2 mg for test substance 1; 10 mg for test substance 2; 0.04 g for test substance 3.

IDENTITY TESTS

Colour and other reactions

1. To test substance 1 add 3 ml of water and 1 drop of ferric chloride (25 g/l)TS; a green colour is produced. Add 10-20 mg of sodium hydrogen carbonate R; a blue colour is produced that changes to red-violet.
2. Add test substance 2 to a mixture of 1.0 ml of sulfuric acid (~5 g/l)TS and 2.0 ml of water; then add 3 ml of ammonium molybdate (95 g/l)TS, mix; a golden yellow colour is produced. While stirring add 2 ml of sodium hydroxide (~200 g/l)TS; a pink coloration is produced.
3. To test substance 3 add 5 ml of water, 2.0 ml of potassio-cupric tartrate TS and heat; the colour of the solution turns green and a red precipitate is produced.

METRONIDAZOLE TABLETS

Description. Each tablet usually contains 200-500 mg of metronidazole.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.10 g of metronidazole.
2. Grind the tablets, weigh out the above calculated equivalent amount to metronidazole as powdered material, shake it with 30 ml of water, filter, evaporate the filtrate to a smaller volume, allow to crystallize, separate the crystals, dry at 105 °C for 1 hour and use the dried material as the test substance. Divide the test substance into three equal parts.

IDENTITY TESTS

Melting behaviour. The test substance melts at about 160 °C with decomposition.

Colour and other reactions

1. To 1 part of the test substance add 0.05 g of 4-dimethylaminobenzaldehyde R dissolved in 2.0 ml of hydrochloric acid (~70 g/l)TS; a yellowish colour is produced. Add 0.05 g of zinc R powder; the colour changes to red-orange.
2. To 1 part of the test substance add 5 ml of sodium hydroxide (~80 g/l)TS and boil; the solution shows the following colours in turn: pink, pink-violet, red-violet, red, red-brown, yellow-brown and yellow.

NEOMYCIN SULFATE TABLETS

Description. Each tablet usually contains 150-350 mg of neomycin sulfate.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.05 g of neomycin sulfate.

2. Grind the tablets, weigh out the above calculated equivalent amount to neomycin sulfate as powdered material, shake it with 10 ml of water, filter and evaporate the filtrate to dryness. Use the residue as the test substance. Divide the test substance into three equal parts.

IDENTITY TESTS

Colour and other reactions

1. Dissolve 1 part of the test substance in 2.0 ml of a solution of 25 mg of sodium acetate R per ml of water, and add 1.0 ml of a freshly prepared solution of 5 mg of triketohydrindene hydrate R per ml of water. Heat the mixture on a water-bath for 5 minutes; a violet colour is produced.
2. Dissolve 1 part of the test substance in about 2 ml of sulfuric acid (~1760 g/l)TS; the solution is colourless to slightly yellow. Add 8 ml of water; no precipitate is observed.
3. Dissolve 1 part of the test substance in 2.0 ml of water, add 5 drops of hydrochloric acid (~70 g/l)TS and 5 drops of barium chloride (50 g/l)TS; a white precipitate is formed.

NITROFURANTOIN TABLETS

Description. Each tablet usually contains 100 mg of nitrofurantoin.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 20 mg and about 1 mg of nitrofurantoin.
2. Grind the tablets, weigh out the above calculated equivalent amounts to nitrofurantoin as powdered material and use them directly: 20 mg for test substance 1; two portions of about 1 mg for test substance 2.

IDENTITY TESTS

Colour and other reactions

1. Suspend test substance 1 in a mixture of 5 ml of sodium hydroxide (~80 g/l)TS and 5 ml of water; an orange-red solution is produced which changes to dark brown.
2. To 1 portion of test substance 2 add 1.0 ml of dimethylformamide R, 2 drops of potassium hydroxide/ethanol TS and shake; a brown colour is produced.
3. Mix the remaining portion of test substance 2 with about 2 ml of sulfuric acid (~1760 g/l)TS; a light yellow colour is produced. Add 10 mg of resorcinol R; the colour changes to strong yellow, then orange and finally to brown.

NYSTATIN TABLETS

Description. Each tablet usually contains 500 000 IU of nystatin. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amount equivalent to 0.16 g of nystatin.
2. Grind the tablets or cores, weigh out the above calculated equivalent amount to nystatin as powdered material and use it directly as the test substance. Divide the test substance into three equal parts.

IDENTITY TESTS

Colour and other reactions

1. To 1 part of the test substance add 2.0 ml of sulfuric acid (~1760 g/l)TS; a brown-violet colour is produced.
2. To 1 part of the test substance add 1.0 ml of ethanol (~750 g/l)TS and about 1 ml of hydrochloric acid (~250 g/l)TS, shake and filter. To the filtrate add a few crystals of resorcinol R, and heat on a water-bath for 2 minutes; a pink colour is produced.
3. To 1 part of the test substance add 2.0 ml of ethanol (~750 g/l)TS, shake and filter. To the filtrate add about 1 ml of hydrochloric acid (~250 g/l)TS, and 2 drops of a solution composed of 1.0 ml of ferric chloride (25 g/l)TS and 10 ml of water; a yellowish green colour is produced.

PARACETAMOL TABLETS

Description. Each tablet usually contains 100-500 mg of paracetamol.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.5 g of paracetamol.
2. Grind the tablets, weigh out the above calculated equivalent amount to paracetamol as powdered material, shake it with 20 ml of hot ethanol (~750 g/l)TS, filter, evaporate the filtrate to dryness on a water-bath and use the residue as the test substance.

IDENTITY TESTS

Melting point. The test substance melts at about 170 °C.

Colour and other reactions

1. Dissolve 0.10 g of the test substance in 10 ml of water and add 0.5 ml of ferric chloride (25 g/l)TS; an intense blue colour is produced.

2. To 0.10 g of the test substance add about 2 ml of hydrochloric acid (~250 g/l)TS and heat to boiling for 1 minute. Following this add 10 ml of water, 1 drop of potassium dichromate (100 g/l)TS, and shake; a violet colour is produced.

3. Dissolve 35 mg of the test substance in 2.0 ml of ethanol (~750 g/l)TS and add 2 ml of 4-dimethylaminobenzaldehyde TS; the solution remains almost colourless. Heat the solution in a water-bath for 5 minutes; a yellow colour is produced.

PHENOBARBITAL TABLETS

Description. Each tablet usually contains 50-100 mg of phenobarbital.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.5 g of phenobarbital.
2. Grind the tablets, weigh out the above calculated equivalent amount to phenobarbital as powdered material, and use it directly as the test substance. Divide the test substance into 5 equal parts.
3. Shake 4 parts of the test substance with 10 ml of dehydrated ethanol R, filter, evaporate the filtrate to dryness and use the residue as test substance 1.
4. Suspend 1 part of the test substance in 5 ml of ethanol (~750 g/l)TS, place a strip of filter-paper into it and allow the solution to ascend for about 4 cm. Take out the strip, cut away the lower dipped portion, as well as the part that has not been wetted by the solution and dry the remaining part of the strip in air at room temperature (test-paper).

IDENTITY TESTS

Melting point. Test substance 1 melts at about 174 °C.

Heating behaviour. Heat test substance 1; a colourless melt is obtained which has a characteristic odour and white fumes are developed. When inflamed, it burns with a strong luminous flame. When ignited, the residue has a yellowish brown colour that turns finally to black.

Colour and other reactions

1. Dissolve 20 mg of test substance 1 in 5 ml of methanol R, add 1 drop of cobalt(II) chloride (30 g/l)TS and 3-4 drops of ammonia (~100 g/l)TS; a violet colour is produced.

Alternate test by filter-paper technique.

Place onto the test-paper 1 drop of cobalt(II) chloride (30 g/l)TS, followed by 1 drop of ammonia (~100 g/l)TS applied over the same spot; a yellowish spot with a violet border is produced.

2. Dissolve 0.10 g of test substance 1 in a mixture of 5 ml of water and 0.5 ml of sodium hydroxide (~80 g/l)TS, filter, then add 1.0 ml of citric acid (90 g/l)TS; a white, voluminous precipitate is produced (distinction from barbital).

3. Using heat dissolve 10 mg of test substance 1 in 10 ml of water. Cool and pour into a mixture composed of 0.5 ml of potassium bromate (15 g/l)TS, 0.05 g of potassium bromide R and 1.0 ml of hydrochloric acid (~70 g/l)TS. Shake; a stable reddish yellow colour is obtained (distinction from hexobarbital).

4. To 0.20 g of test substance 1 add about 2 ml of sulfuric acid (~1760 g/l)TS and 20 mg of sodium nitrate R; a yellow coloration is produced.

PREDNISOLONE TABLETS

Description. Each tablet usually contains 5.0 mg of prednisolone.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.10 g of prednisolone.
2. Grind the tablets, weigh out the above calculated equivalent amount to prednisolone as powdered material, shake it with 25 ml of chloroform R and filter. Evaporate the filtrate to dryness on a water-bath and use the residue as the test substance.

IDENTITY TESTS

Colour and other reactions

1. To about 2 mg of the test substance add about 2 ml of sulfuric acid (~1760 g/l)TS and allow to stand for 5 minutes; the following colours are observed in turn, yellow, orange, pink and light red. Following this dilute the solution very cautiously with 10 ml of water; a yellow colour is first observed changing to colourless and after a few minutes a light brown flocculent precipitate is produced.
2. To about 2 mg of the test substance add about 1 ml of phosphoric acid (~1440 g/l)TS and heat cautiously; the following colours are observed in turn, yellow, green, orange and reddish brown.
3. To 0.05 g of the test substance add 0.5 ml of potassium hydroxide/ethanol TS, 1.5 ml of ethanol (~750 g/l)TS and heat in a water-bath for 5 minutes. Cool, cautiously add 1.0 ml of water and 1 ml of sulfuric acid (~1760 g/l)TS and heat gently for 1 minute; no odour of ethyl acetate is perceptible (distinction from prednisolone acetate).
4. To 5 mg of the test substance add 2.0 ml of ethanol (~750 g/l)TS and shake. Following this add 1.0 ml of potassium-cupric tartrate TS and heat to boiling; an orange precipitate is slowly formed.

PRIMAQUINE DIPHOSPHATE TABLETS

Description. Each tablet usually contains 7.5-15 mg of primaquine diphosphate. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amount equivalent to 0.05 g of primaquine diphosphate.
2. Grind the tablets or cores, weigh out the above calculated equivalent amount to primaquine diphosphate as powdered material, suspend it in 6 ml of water and filter. Place 2 strips of filter-paper into the filtrate and allow the solution to ascend for about 4 cm. Take out the strip, cut away the lower dipped portion as well as the part that has not been wetted by the solution and dry the remaining part of the strip in air at room temperature (test-paper). Keep the remaining filtrate as the test solution.

IDENTITY TEST

Colour and other reactions

1. Place onto a test-paper 1 drop of ceric ammonium sulfate/nitric acid TS; a deep violet spot is produced, which gradually disappears (distinction from chloroquine).
2. Place onto a test-paper 1 drop of gold chloride TS; a violet spot is at once produced.
3. To the remaining test solution add 2.0 ml of sodium hydroxide (~80 g/l)TS and filter. Neutralize the filtrate with sulfuric acid (~100 g/l)TS and add 10 ml of ammonium molybdate (95 g/l)TS; a yellowish precipitate is formed which is soluble in ammonia (~100 g/l)TS and in nitric acid (~130 g/l)TS.

PROCAINAMIDE HYDROCHLORIDE TABLETS

Description. Each tablet usually contains 250-500 mg of procainamide hydrochloride.

Preparation of the sample

1. Weigh 1 tablet and calculate the amounts equivalent to 10 mg and 0.07 g of procainamide hydrochloride.
2. Grind the tablets, weigh out the above calculated equivalent amounts to procainamide hydrochloride as powdered material and use them directly; two portions of 10 mg for test substance 1; 0.07 g for test substance 2.

IDENTITY TESTS

Colour and other reactions

1. To 1 portion of test substance 1 add 1.0 ml of water, 5 drops of hydrochloric acid (~70 g/l)TS, 0.5 ml of sodium nitrite (10 g/l)TS, 1.0 ml of sodium hydroxide (~80 g/l)TS and 5 mg of 2-naphthol R; an orange-red solution with a red precipitate are produced.
2. To test substance 2 add 1.0 ml of water, 1.0 ml of potassium ferrocyanide (45 g/l)TS, 0.5 ml of hydrochloric acid (~70 g/l)TS, and heat to boiling; a dark green precipitate is produced.

3. To the remaining portion of test substance 1 add 2.0 ml of water, shake and filter. To the filtrate add a few drops of silver nitrate (40 g/l)TS; a white, curdy precipitate is produced, which is insoluble in nitric acid (~130 g/l)TS, but soluble in an excess of ammonia (~100 g/l)TS.

PROMETHAZINE HYDROCHLORIDE INJECTION

Description. The injection is a sterile solution usually containing 25 mg of promethazine hydrochloride in 1.0 ml of a suitable vehicle.

Preparation of the sample

Pool the contents of the ampoules equivalent to 30 mg of promethazine hydrochloride and use it directly as the test solution. Divide the test solution into three equal volumes.

IDENTITY TESTS

Colour and other reactions

1. Evaporate 1 volume of the test solution to dryness on a water-bath, dissolve the residue in 5 ml of sulfuric acid (~1760 g/l)TS and allow to stand for 5 minutes; a red colour is produced. Add a few drops of potassium dichromate (100 g/l)TS; the solution is almost discoloured (distinction from promazine).
2. Shake 1 volume of the test solution with 5 ml of chloroform R, filter, to the filtrate add 1.0 ml of sodium metaperiodate (60 g/l)TS and 2.0 ml of sulfuric acid (~100 g/l)TS. Shake vigorously and allow the layers to separate; the aqueous layer remains colourless where as the chloroform layer shows a dark green colour (distinction from chlorpromazine).
3. To the remaining volume of the test solution add 5 ml of water, shake well and filter. To the filtrate add 4-5 drops of nitric acid (~1000 g/l)TS; a dark red colour is produced which suddenly fades to almost colourless. Add 2.0 ml of silver nitrate (40 g/l)TS; a white, curdy precipitate is produced.

PROMETHAZINE HYDROCHLORIDE TABLETS

Description. Each tablet usually contains 10-25 mg of promethazine hydrochloride. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amount equivalent to 30 mg of promethazine hydrochloride.
2. Grind the tablets, weigh out the above calculated equivalent amount to promethazine hydrochloride as powdered material and use it directly as the test substance. Divide the test substance into three equal parts.
3. Shake 1 part of the test substance with 5 ml of chloroform R, filter and use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To 1 part of the test substance add 5 ml of sulfuric acid (~1760 g/l)TS and allow to stand for 5 minutes; a red colour is produced. Add a few drops of potassium dichromate (100 g/l)TS; the solution is almost discolored (distinction from promazine).
2. To the test solution add 1.0 ml of sodium metaperiodate (60 g/l)TS and 2.0 ml of sulfuric acid (~100 g/l)TS. Shake vigorously and allow the layers to separate; the aqueous layer remains colourless whereas the chloroform layer shows a dark green colour (distinction from chlorpromazine).
3. To the remaining part of the test substance add 5 ml of water, shake well and filter. To the filtrate add 4-5 drops of nitric acid (~1000 g/l)TS; a dark red colour is produced which suddenly fades to almost colourless. Add 2.0 ml of silver nitrate (40 g/l)TS; a white, curdy precipitate is produced.

PROPRANOLOL HYDROCHLORIDE TABLETS

Description. Each tablet usually contains 10-40 mg of propranolol hydrochloride.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.20 g of propranolol hydrochloride.
2. Grind the tablets, weigh out the above calculated equivalent amount to propranolol hydrochloride as powdered material, triturate it with 10 ml of water, filter and use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. Evaporate to dryness 1 drop of the test solution on a water-bath, to the residue add 1 drop of fuming nitric acid R; a deep purple colour is produced. Evaporate again to dryness; a greenish yellow colour is obtained. Moisten the residue with freshly prepared potassium hydroxide/ethanol TS; the colour of the mixture changes to orange.
2. To 2.0 ml of the test solution add 1-2 drops of nitric acid (~130 g/l)TS and 4-5 drops of silver nitrate (40 g/l)TS; a white, curdy precipitate is obtained, which after being well washed with water, is soluble in ammonia (~100 g/l)TS.
3. After redering the remaining test solution alkaline with sodium hydroxide (~200 g/l)TS, extract it with three quantities, each of 10 ml of chloroform R. Wash the combined extracts with water until the washings are free from alkali. Dry the chloroform extracts with anhydrous sodium sulfate R, filter and evaporate the filtrate to dryness. Dry the residue under reduced pressure at 50 °C for 1 hour; melting point, about 94 °C.

PYRIDOXINE HYDROCHLORIDE TABLETS

Description. Each tablet usually contains 25 mg of pyridoxine hydrochloride.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 30 mg of pyridoxine hydrochloride.
2. Grind the tablets, weigh out the above calculated equivalent amount to pyridoxine hydrochloride as powdered material and use it directly as the test substance. Divide the test substance into 3 equal parts.
3. Shake 2 parts of the test substance with 5 ml of water, filter and use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To 1.0 ml of the test solution add 1.0 ml of ferric chloride (25 g/l)TS; a red-brown colour is produced. Add 2.0 ml of hydrochloric acid (~70 g/l)TS; the colour of the solution turns to yellow.
2. To 0.5 ml of sulfanilic acid TS add 3 drops of sodium nitrite (10 g/l)TS, 1.0 ml of sodium hydroxide (~80 g/l)TS and 1 part of the test substance; a golden yellow colour appears after 2 minutes. Add 2 ml of acetic acid (~300 g/l)TS; the colour of the solution turns to orange.
3. To 2.0 ml of the test solution add 0.5 ml of nitric acid (~130 g/l)TS and 1.0 ml of silver nitrate (40 g/l)TS; a white precipitate is produced. Add 3-4 ml of ammonia (~100 g/l)TS; the precipitate dissolves.

QUINIDINE SULFATE TABLETS

Description. Each tablet usually contains 200 mg of quinidine sulfate.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 150 mg of quinidine sulfate.
2. Grind the tablets, weigh out the above calculated equivalent amount to quinidine sulfate as powdered material and use it directly as the test substance. Divide the test substance into 3 equal parts.
3. Shake 2 parts of the test substance with 10 ml of water, filter, and use the filtrate as the test solution.
4. Suspend 1 part of the test substance in 5 ml of ethanol (~750 g/l)TS, place a strip of filter-paper into the suspension and allow the solution to ascend for about 4 cm. Take out the strip, cut away the lower dipped portion as well as the part that has not been wetted by the solution, and dry the remaining part of the strip in air at room temperature (test-paper).

IDENTITY TESTS

Colour and other reactions

1. The test solution produces a slight blue fluorescence. To 1.0 ml of the test solution add a few drops of sulfuric acid (~100 g/l)TS and dilute to 5 ml with water; in ultraviolet light (254 nm) a vivid blue fluorescence is observed.

Alternate test by filter-paper technique:

Place onto the test-paper 1 drop of sulfuric acid (~100 g/l)TS and observe it in ultraviolet light (254 nm); a vivid blue fluorescent spot is produced.

2. To about 0.5 ml of the test solution add 4 ml of water, 1 drop of bromine TS and 1.0 ml of ammonia (~100 g/l)TS; a bluish green solution is slowly produced.

3. To 3 ml of the test solution add 5 ml of water, 0.10 g of potassium iodide R and shake; a white precipitate is formed (distinction from quinine).

4. To 3 ml of the test solution add 0.5 g of potassium sodium tartrate R, and shake; the solution remains unchanged (distinction from quinine).

5. To 1.0 ml of the test solution add a few drops of hydrochloric acid (~70 g/l)TS and 1.0 ml of barium chloride (50 g/l)TS; a white precipitate is produced.

RESERPINE TABLETS

Description. Each tablet usually contains 0.10-0.25 mg of reserpine.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 3 mg of reserpine.
2. Grind the tablets, weigh out the above calculated equivalent amount to reserpine as powdered material and use it directly as the test substance. Divide the test substance into three equal parts.

IDENTITY TESTS

Colour and other reactions

1. Mix 1 part of the test substance with 5 mg of 4-dimethylaminobenzaldehyde R and 4 drops of glacial acetic acid R. After this add 4 drops of sulfuric acid (~1760 g/l)TS; a green colour is produced. Further add about 1 ml of glacial acetic acid R; the colour turns to red.

2. Suspend the remaining parts of the test substance in 3.0 ml of ethanol (~750 g/l)TS, shake and filter. To the filtrate add 1 drop of sulfuric acid (~100 g/l)TS and a few drops of sodium nitrite (10 g/l)TS; the colour of the solution turns slowly to yellowish green and it has a greenish fluorescence.

RETINOL ORAL SOLUTION

Description. The solution usually contains retinol acetate or retinol palmitate in a suitable vegetable oil, equivalent to 15 mg of retinol per ml or 50 000 IU per ml.

Preparation of the sample

Pool the contents of the containers equivalent to 5 mg of retinol acetate or retinol palmitate and use it directly as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To about 2 ml of sulfuric acid (~1760 g/l)TS add 1 drop of the test solution; a deep purple colour is produced. Allow to stand for a few minutes; the colour changes to wine-red.
2. Mix 1 drop of the test solution with 5 ml of chloroform R, add about 0.5 ml of acetic anhydride R, 2 drops of sulfuric acid (~1760 g/l)TS, and shake well; the yellowish colour of the solution changes to dark blue.

RIBOFLAVIN TABLETS

Description. Each tablet usually contains 5 mg of riboflavin. The tablets may be coated.

Preparation of the sample

1. In the event that tablets are coated, carefully remove the coating by scraping. Weigh 1 tablet or core and calculate the amount equivalent to 25 mg of riboflavin.
2. Grind the tablets or cores, weigh out the above calculated equivalent amount to riboflavin as powdered material and use it directly as the test substance. Divide the test substance into five equal parts.

IDENTITY TESTS

Colour and other reactions

1. To 1 part of test substance add 10 ml of hot water and shake; a yellow suspension is produced with a green fluorescence. Add a few drops of hydrochloric acid (~70 g/l)TS or a few drops of sodium hydroxide (~80 g/l)TS; the fluorescence disappears.
2. To 3 parts of the test substance add about 2 ml of sulfuric acid (~1760 g/l)TS and shake; a deep red colour is produced.
3. To the remaining part of test substance add 2.0 ml of silver nitrate (40 g/l)TS; after a few minutes an orange colour is produced. Allow to stand for a few hours; a red precipitate is formed.

SALBUTAMOL SULFATE TABLETS

Description. Each tablet usually contains 4.0 mg of salbutamol sulfate.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 25 mg of salbutamol sulfate.
2. Grind the tablets, weigh out the above calculated equivalent amount to salbutamol sulfate as powdered material, shake it with 10 ml of water, filter and use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To 4 ml of the test solution add 0.10 ml of ferric chloride (25 g/l)TS; a reddish violet colour develops. Add 10 mg of sodium hydrogen carbonate R; a fleshy precipitate is produced with an evolution of gas. Add 1-2 drops of sulfuric acid (~1760 g/l)TS; the solution becomes colourless.
2. To 2.0 ml of the test solution add 0.5 ml of barium chloride (50 g/l)TS; a white precipitate is produced.
3. To 2.0 ml of the test solution add 2-3 drops of sulfuric acid (~100 g/l)TS and 2-3 drops of potassium permanganate (10 g/l)TS; the colour of permanganate is discharged.

SALICYLIC ACID LOTION

Description. The solution usually contains 50 mg of salicylic acid in 1.0 ml of a suitable vehicle.

Preparation of the sample

1. Pool the contents of the containers equivalent to 0.50 g of salicylic acid and use it directly as the test solution. Divide the test solution into five equal volumes.
2. Evaporate 2 volumes of the test solution to dryness in a current of air and use the residue as the test substance.
3. Place a strip of filter-paper into 2 volumes of the test solution and allow the solution to ascend for about 4 cm. Take out the strip, cut away the lower dipper portion as well as the part that has not been wetted by the solution and dry the remaining part of the strip in air at room temperature (test-paper).

IDENTITY TESTS

Heating behaviour. Heat the test substance in a dry test-tube; the substance melts, sublimes (collect the sublimate) and finally boil; the initial pungent odour changes to an intense phenolic odour.

Melting point of the sublimate. About 158 °C.

Colour and other reactions

To half of 1 volume of the test solution add 5 ml of water and warm gently. Insert a strip of pH-indicator paper R into the solution; its coloration is changed to an acidic range. To the cooled solution add a few drops of ferric chloride (25 g/l)TS; a dark violet colour is produced.

Alternate test by filter-paper technique:

Place onto the test-paper 1 drop of ferric chloride (25 g/l)TS; a strong violet spot is produced.

SODIUM CITRATE SOLUTION

Description. The solution usually contains 32.0 mg of sodium citrate dihydrate in 1.0 ml of a suitable vehicle (equivalent to 38.0 mg of sodium citrate pentahydrate).

Preparation of the sample

Pool the contents of the containers equivalent to about 0.3 g of sodium citrate and use it directly as the test solution.

IDENTITY TESTS

Colour and other reactions

1. Introduce the test solution from a nichrome or platinum wire sealed to a glass rod into a nonluminous flame; a strong yellow colour can be observed.
2. Acidify 2.0 ml of the test solution with acetic acid (~300 g/l)TS, add 2.0 ml of magnesium uranyl acetate TS or zinc uranyl acetate TS and scratch the sides of the tubes to induce crystallization; a light yellow, crystalline precipitate is produced.
3. To 5 ml of the test solution add 3 ml of mercuric chloride (65 g/l)TS and heat to boiling. While boiling add a few drops of potassium permanganate (10 g/l)TS; the violet colour is immediately discharged and a white precipitate is produced.
4. The test solution is alkaline when tested with pH-indicator paper R.

SODIUM HYDROGEN CARBONATE TABLETS

Description. Each tablet usually contains 325-650 mg of sodium hydrogen carbonate.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.20 g of sodium hydrogen carbonate.
2. Grind the tablets, weigh out the above calculated equivalent amount to sodium hydrogen carbonate as powdered material and use it directly as the test substance. Keep a small amount of the test substance for test 1 and divide the remaining material into two equal parts.

IDENTITY TESTS

Colour and other reactions

1. Moisten the small amount of test substance set aside with a few drops of hydrochloric acid (~70 g/l)TS and introduce it from a nichrome or platinum wire sealed to a glass rod into a nonluminous flame; a bright yellow colour appears in the flame.
2. To 1 part of the test substance add 5 ml of a water and 2 drops of phenolphthalein/ethanol TS; a pink colour is produced. Heat to boiling; a gas is evolved and the colour of the solution turns to red-violet.
3. To the remaining part of the test substance add about 1 ml of acetic acid (~300 g/l)TS; a gas evolves which is colourless and odourless. Pass the generated gas into 5 ml of calcium hydroxide TS; a white precipitate is formed immediately.

SODIUM NITRITE INJECTION

Description. The injection is a sterile solution usually containing 30 mg of sodium nitrite in 1.0 ml of a suitable vehicle.

Preparation of the sample

Pool the contents of the ampoules equivalent to 0.3 g of sodium nitrite and use it directly as the test solution. Divide the test solution into four equal volumes.

IDENTITY TESTS

Colour and other reactions

1. Introduce the test solution from a nichrome or platinum wire sealed to a glass rod into a nonluminous flame; a strong yellow colour can be observed.
2. Acidify 1 volume of the test solution with acetic acid (~300 g/l)TS and add 2.0 ml of magnesium uranyl acetate TS or zinc uranyl acetate TS; a light yellow, crystalline precipitate is produced.
3. To 1 volume of the test solution add 20 mg of ferrous sulfate R and 1.0 ml of sulfuric acid (~100 g/l)TS; a gas is evolved and the solution acquires a strong brown-green colour.
4. To 1 volume of the test solution add 2.0 ml of hydrochloric acid (~70 g/l)TS; nitrous vapours are produced.
5. The test solution is neutral when tested with pH-indicator paper R.

STREPTOMYCIN SULFATE POWDER FOR INJECTION

Description. Each vial contains a sterile powder usually equivalent to 1.0 g of streptomycin sulfate.

Preparation of the sample

1. Weigh the contents of 1 vial and calculate the amounts equivalent to 10 mg and 50 mg of streptomycin sulfate.
2. Empty the vials, weigh out the above calculated equivalent amounts to streptomycin sulfate and use them directly; three portions of 10 mg for test substance 1; three portions of 50 mg for test substance 2.

IDENTITY TESTS

Colour and other reactions

1. Dissolve 2 portions of test substance 2 in 1.0 ml of sodium hydroxide (~80 g/l)TS and heat on a water-bath for 5 minutes. Cool, add 1.5 ml of hydrochloric acid (~70 g/l)TS and 3 drops of ferric chloride (25 g/l)TS; an intense violet colour is produced.
2. Dissolve the remaining portion of test substance 2 in 1.0 ml of pyridine R, add 1.0 ml of sodium hydroxide (~80 g/l)TS, 3 drops of benzenesulfonyl chloride R and shake well; a violet colour is produced.
3. Dissolve 1 portion of test substance 1 in 2.0 ml of 4-dimethylaminobenzaldehyde TS and heat on water-bath for 2 minutes; a purplish red colour is produced.
4. Dissolve the remaining portions of test substance 1 in 2.0 ml of water, add 3 drops of barium chloride (50 g/l)TS; a white, crystalline precipitate is produced.

SULFACETAMIDE SODIUM OPHTHALMIC SOLUTION

Description. The solution usually contains 100 mg of sulfacetamide solution in 1.0 ml of a suitable vehicle.

Preparation of the sample

Pool the contents of the containers equivalent to 0.15 g of sulfacetamide sodium and use it directly as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To the test solution add 2.0 ml of acetic acid (~60 g/l)TS; a white precipitate is produced. Collect the precipitate on a filter-paper, wash with a minimum amount of water and dry; melting point, about 183 °C. (Retain the crystals for tests 2, 3 and 4 and divide them in three equal parts).
2. Dissolve 1 part of the crystals obtained in test 1 in 2.0 ml of warm hydrochloric acid (~70 g/l)TS and cool in ice. Add 2-3 drops of sodium nitrite (10 g/l)TS, allow to stand for a few minutes, add 1.0 g of urea R and a solution of 10 mg of 2-naphthol R dissolved in 2.0 ml of sodium hydroxide (~80 g/l)TS; an orange-red precipitate is produced.

3. To 1 part of the crystals obtained in test 1 add 5 drops of ethanol (~750 g/l)TS, 5 drops of sulfuric acid (~1760 g/l)TS and heat gently; a faint odour of ethyl acetate is perceptible.
4. Acidify the remaining part of the crystals obtained in test 1 with acetic acid (~300 g/l)TS, and add 2.0 ml of magnesium uranyl acetate TS or zinc uranyl acetate TS; a light yellow, crystalline precipitate is formed.

SULFADIMIDINE TABLETS

Description. Each tablet usually contains 500 mg of sulfadimidine.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 0.20 g of sulfadimidine.
2. Grind the tablets, weigh out the above calculated equivalent amount to sulfadimidine as powdered material and use it directly as the test substance. Divide the test substance into eight equal parts.

IDENTITY TESTS

Heating behaviour. Heat a small quantity of the test substance; it melts, becomes yellow and a characteristic odour with the evolution of white fumes is produced. Apply intense heat; it turns brown, then black and when inflamed, it burns with a luminous flame.

Colour and other reactions

1. To 2 parts of the test substance add 1.0 ml of hydrochloric acid (~250 g/l)TS and 1.5 ml of water. Shake, filter and to the filtrate add 1 ml of formaldehyde TS; no precipitate is produced. Boil the mixture; it becomes yellow, after cooling a precipitate separates. Add 4 ml of sodium hydroxide (~80 g/l)TS; the precipitate remains undissolved.
2. To 1 part of the test substance add 1.0 ml of a mixture composed of 0.5 ml of sodium hydroxide (~80 g/l)TS and 9.5 ml of water. Shake and add 2 drops of copper(II) sulfate (160 g/l)TS; a precipitate is formed the colour of which changes quickly from yellow to light green. Shake the mixture; the colour of the precipitate turns quickly to light brown.
3. To 3 parts of the test substance add 1.0 ml of water and 1.0 ml of 4-dimethylaminobenzaldehyde TS; a yellow-orange colour is produced.

TETRACYCLINE HYDROCHLORIDE CAPSULES

Description. Each capsule usually contains 250 mg of tetracycline hydrochloride.

Preparation of the sample

1. Weigh the contents of 1 capsule and calculate the amount equivalent to 0.10 g of tetracycline hydrochloride.

2. Empty the capsules and weigh out the above calculated equivalent amount to tetracycline hydrochloride, shake it with 10 ml of water, filter and use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To about 2 ml of sulfuric acid (~1760 g/l)TS add 2 drops of the test solution; a purple violet colour is produced which remains unchanged for more than 2 minutes. Allow to stand for 5 minutes, then cautiously add 2.0 ml of water; a yellow colour is produced.
2. In a porcelain dish warm 2.0 ml of zinc chloride (500 g/l)TS until a skin is formed on the surface of the solution. Following this add 2 drops of the test solution and continue to warm for 1 minute; a yellow-orange colour is produced.
3. To 1.0 ml of the test solution add a few drops of nitric acid (~130 g/l)TS and a few drops of silver nitrate (40 g/l)TS; a white, curdy precipitate is produced.

THIAMINE HYDROCHLORIDE TABLETS

Description. Each tablet usually contains 50 mg of thiamine hydrochloride.

Preparation of the samples

1. Weigh 1 tablet and calculate the amount equivalent to 0.05 g of thiamine hydrochloride.
2. Grind the tablets, weigh out the above calculated equivalent amount to thiamine hydrochloride as powdered material, shake it with 5 ml of water, filter and use the filtrate as the test solution.

IDENTITY TESTS

Colour and other reactions

1. To 1.0 ml of the test solution add 1.0 ml of sodium hydroxide (~80 g/l)TS, 4 drops of potassium ferricyanide (50 g/l)TS, 5 ml of 2-butanol R and shake. Allow the layers to separate; the 2-butanol layer shows a blue fluorescence in bright daylight or in ultraviolet light (365 nm) which after the addition of acid disappears and recurs when the solution is made alkaline.
2. To 1.0 ml of the test solution add 1-2 drops of nitric acid (~130 g/l)TS and a few drops of silver nitrate (40 g/l)TS; a white, curdy precipitate is produced. Add a few drops of ammonia (~100 g/l)TS, and heat; the precipitate dissolves and the solution turns yellow, then brown and on standing a brown turbidity is produced.

TRIMETHOPRIM TABLETS

Description. Each tablet usually contains 80-300 mg of trimethoprim.

Preparation of the sample

1. Weigh 1 tablet and calculate the amount equivalent to 20 mg of trimethoprim.
2. Grind the tablets, weigh out the above calculated equivalent amount to trimethoprim as powdered material, shake it with 5 ml of chloroform R, filter, evaporate the filtrate to dryness and use the residue as the test substance.

IDENTITY TESTS

Colour and other reactions

To the test substance add 5 ml of sulfuric acid (~1760 g/l)TS, 1 drop of ferric chloride (25 g/l)TS and warm the solution in a water-bath for 3 minutes. Cool, add to the yellow solution 1 drop of nitric acid (~130 g/l)TS; the colour of the solution turns red.

6. REAGENTS

The preparation of the required reagents is described in "Basic tests for pharmaceutical substances." A list of additional reagents is as follows:

Acetic Acid (~60 g/l)TS

Calcium oxide R

Cyclohexane R

Hydrochloric acid (0.05 mol/l)VS

Hydroxylamine hydrochloride (10 g/l)TS

Potassium bicarbonate R

Sodium chloride R

Sodium hydrogen carbonate (40 g/l)TS

Sodium nitrate R

Tannic acid R

Tannic acid (100 g/l)TS

Trinitrophenol, alkaline, TS

Procedure: Mix 20 ml of a 10 mg/ml solution of trinitrophenol R with 10 ml of a 50 mg/ml solution of sodium hydroxide R, dilute with water to 100 ml and mix.

Note: Do not use alkaline trinitrophenol TS longer than 48 hours after preparation.

Zinc uranyl acetate TS

Procedure: Dissolve 5 g of uranyl acetate R in a mixture of 1.5 ml of glacial acetic acid R and water, and dilute to 50 ml with water. Then dissolve 15 g of zinc acetate R in a mixture of 1.5 ml of glacial acetic acid R and water, and dilute to 50 ml with water. Mix the two solutions, allow to stand overnight, and filter through a dry filter, if necessary.

ANNEX

TESTS THAT REQUIRE FINAL VALIDATION OR IMPROVEMENT

In the process of verification several tests were modified according to suggestions from collaborators. Additional work is needed with regard to certain products as indicated below:

Amitriptyline hydrochloride tablets

- The extraction should be changed avoiding ether as a solvent.
- The replacement of the m.p. test by an alternative is desirable.

Betamethasone valerate cream

Tests 2 and 3 need validation.

Chloramphenicol palmitate oral suspension

Validation is needed.

Chlorpromazine hydrochloride tablets

Tests 3 was modified and needs validation.

Cyclophosphamide tablets

Problems were noted with test 1, a different extraction may be considered.

Diethylcarbamazine dihydrogen citrate tablets

Needs validation.

Ibuprofen tablets

Test 2, thionyl chloride is a hazardous reagent; a different test is desirable.

Isoprenaline sulfate tablets

Needs validation.

Phenobarbital tablets

Test 4 needs validation.

Prednisolone tablets

Needs validation.

Trimethoprim tablets

There is only 1 test, an additional one is desirable.

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