

**HEALTHCARE MINISTRY OF UKRAINE
NATIONAL PHARMACEUTICAL UNIVERSITY OF PHARMACY
PHARMACEUTICAL CHEMISTRY DEPARTMENT**

**Journal for laboratory work
in Pharmaceutical chemistry
4^d year, 7th semester student
speciality «Pharmacy»**

_____ **group**

Kharkov

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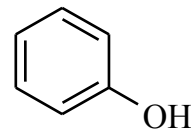
GENERAL LABORATORY SAFETY PROCEDURES AND RULES

1. All students must read and understand the information in this document with regard to laboratory safety, fire precaution and emergency procedures prior to the first laboratory session.
2. Keep the work area clear of all materials except those needed for your work. Students must keep clean and maintain order in the laboratory. Coats should be hung in the hall or placed in a locker.
3. Students should hold to usage instructions of protective equipment. Wear laboratory coats and special laboratory hat.
4. Learn properties of compounds that are used and produced before start working.
5. Execution of a laboratory task is allowed after corresponding theoretical preparation that level is controlled by a lecturer at the beginning of studies. Students must know a corresponding material using texts of lectures and referenced literature.
6. There are chemicals at laboratory benches, and chemical dishes in a special cabinet. Concentrated acids and volatile compounds are stored in fume hoods.
7. Carefully check all equipment, apparatus and dishes before begin working. Make sure that all installations and equipment are correctly built and all chemicals are qualified.
8. Carrying our experiment in dirty dishes is forbidden. Chemical dishes should be washed after execution of a laboratory task immediately.
9. Never eat, drink, or smoke while working in the laboratory.
10. Do not leave operational laboratory devices and equipment unattended.
11. Do not taste any compounds.
12. Do not bend over the dish with heating liquid; do not turn that dish's opening to yourself and other persons. Using unlabeled substances is categorically forbidden.
13. Determine an odour carefully, direct evaporation to yourself with a subtle movement and do not inhale with full breast.
15. Use volatile (fuming acids) and flammable compounds only in a fume hood. Procedures that produce aerosols should be performed in a hood to prevent inhalation of hazardous material.
16. Work with hazardous and poison materials with great care. (salts of barium, lead, mercury, arsenic, copper, metallic mercury, hydrogen sulfide, cyanides), prevent getting to the organism.
17. Conduct all vacuum operations in appropriate dishes behind a table shield or in a fume hood and always wear safety glasses, lab coat, and gloves.
18. Do not pour out acid, alkali, flammable compounds remains to a sink. Do not throw solid rubbish to a sink. Acid, alkali, organic solvents, reagents containing poison materials and valuable metals should be collected in special bottles.
19. Do not take out reagents from a laboratory. Never do unauthorized experiments.
20. Clean up your work area before leaving, switch off all equipment, gas, water, electricity and lock the door.
21. Keep in mind: cleanness, accuracy and laborious task mean successful realization of a laboratory task.

First aid rules

1. Keep to the all safety precautions in accordance with rules and instructions during working in a laboratory.
2. In case of thermal burns a long lotion of a potassium permanganate solution or a compress of an alcohol solution of tannin should be done. In case of cut a wound should be treated with an alcohol solution of iodine and bandaged.
3. Skin, mucous tunic and eyes burns caused by acids should be washed with water first (during 10-15 min), then with 2% solution of potassium hydrocarbonate and then with water again.
4. Burns caused by alkalis should be well washed with water, then with 1% solution of acetic or citric acid.
5. Burns caused by phenol, bromine and such other caustics should be washed with a great amount of 40 % ethanol and lubricated with prescribed ointment.
6. In case of chlorine, bromine, nitrogen oxides and such other compounds poisoning ammonia should be inhaled then go outside, drink milk.
7. In case of badly burns, wounds and poisoning the first aid is given and injured person is sent to an emergency room immediately.

**PHENOL
(PHENOLUM)**



Hydroxybenzene
C₆H₆O

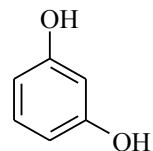
M.M. 94.1

Phenol contains not less than 99.0 per cent and not more than the equivalent of 100.5 per cent of C₆H₆O.

CHARACTERS		
Colourless or faintly pink or faintly yellowish crystals or crystalline masses, deliquescent, soluble in water, very soluble in alcohol, in glycerol and in methylene chloride.		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
IDENTIFICATION		
A Dissolve 0.5 g in 2 ml of <i>concentrated ammonia R</i> . The substance dissolves completely. Dilute to about 100 ml with <i>water R</i> . To 2 ml of the dilute solution add 0.05 ml of <i>strong sodium hypochlorite solution R</i> . A blue colour develops and becomes progressively more intense.		
B. To 1 ml of solution S (see Tests) add 10 ml of <i>water R</i> and 0.1 ml of <i>ferric chloride solution R1</i> . A violet colour is produced which disappears on addition of 5 ml of <i>2-propanol R</i> .		
C. To 1 ml of solution S add 10 ml of <i>water R</i> and 1 ml of <i>bromine water R</i> . A pale-yellow precipitate is formed.		
TESTS		
Solution S. Dissolve 1.0 g in <i>water R</i> and dilute to 15 ml with the same solvent.		
Appearance of solution. Solution S is clear (2.2.1) and not more intensely coloured than reference solution B ₆ (2.2.2, Method II).		
Residue on evaporation. Not more than 0.05 per cent, determined by evaporating 5.000 g to dryness on a water-bath and drying the residue at 100 °C to 105 °C for 1 h.		
Acidity. To 2 ml of solution S add 0.05 ml of <i>methyl orange solution R</i> . The solution is yellow..		

<p>ASSAY</p> <p>Dissolve 2.000 g in <i>water R</i> and dilute to 1000.0 ml with the same solvent. Transfer 25.0 ml of the solution to a ground-glass-stoppered flask and add 50.0 ml of 0.0167 M <i>bromide-bromate</i> and 5 ml of <i>hydrochloric acid R</i>, close the flask, allow to stand with occasional swirling for 30 min and then allow to stand for a further 15 min. Add 5 ml of a 200 g/l solution of <i>potassium iodide R</i>, shake and titrate with 0.1 M <i>sodium thiosulphate</i> until a faint yellow colour remains. Add 0.5 ml of <i>starch solution R</i> and 10 ml of <i>chloroform R</i> and continue the titration with vigorous shaking. Carry out a blank titration. 1 ml of 0.0167 M <i>bromide-bromate</i> is equivalent to 1.569 mg of C₆H₆O.</p>	<p style="text-align: center;">% =</p>	<p>Quantitative content =</p> <p style="text-align: center;">$m_s =$ $V_{Na_2S_2O_3} =$</p>
<p style="text-align: center;">Conclusions</p>		

RESORCINOL
(RESORCINOLUM)



benzene-1,3-diol, calculated
 $C_6H_6O_2$

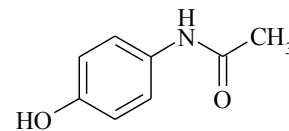
M.M. 110.1

Resorcinol contains not less than 98.5 per cent and not more than the equivalent of 101.0 per cent of benzene-1,3-diol, calculated with reference to the dried substance

CHARACTERS		
A colourless or slightly pinkish-grey, crystalline powder or crystals, turning red on exposure to light and air, very soluble in water and in alcohol, freely soluble in ether.		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
IDENTIFICATION		
A. Melting point (2.2.14): 109 °C to 112 °C.		
B. Dissolve 0.1 g in 1 ml of <i>water R</i> , add 1 ml of <i>strong sodium hydroxide solution R</i> and 0.1 ml of <i>chloroform R</i> , heat and allow to cool. An intense, deep-red colour develops which becomes pale yellow on the addition of a slight excess of hydrochloric acid <i>R</i> .		
C. Thoroughly mix about 10 mg with about 10 mg of <i>potassium hydrogen phthalate R</i> , both finely powdered. Heat over a naked flame until an orange-yellow colour is obtained. Cool and add 1 ml of <i>dilute sodium hydroxide solution R</i> and 10 ml of <i>water R</i> and shake to dissolve. The solution shows an intense green fluorescence		
TESTS		
Solution S. Dissolve 2.5 g in <i>carbon dioxide-free water R</i> and dilute to 25 ml with the same solvent.		

<p>Acidity or alkalinity. To 10 ml of solution S add 0.05 ml of <i>bromophenol blue solution R2</i>. Not more than 0.05 ml of 0.1 M hydrochloric acid or 0.1 M sodium hydroxide is required to change the colour of the indicator.</p>		
<p>ASSAY Dissolve 0.500 g in <i>water R</i> and dilute to 250.0 ml with the same solvent. To 25.0 ml of the solution in a ground-glass-stoppered flask add 1.0 g of <i>potassium bromide R</i>, 50.0 ml of 0.0167 M <i>potassium bromate</i>, 15 ml of <i>chloroform R</i> and 15.0 ml of <i>hydrochloric acid R1</i>. Stopper the flask, shake and allow to stand in the dark for 15 min, shaking occasionally. Add 10 ml of a 100 g/l solution of <i>potassium iodide R</i>, shake thoroughly, allow to stand for 5 min and titrate with 0.1 M <i>sodium thiosulphate</i>, using 1 ml of <i>starch solution R</i> as indicator. 1 ml of 0.0167 M <i>potassium bromate</i> is equivalent to 1.835 mg of C₆H₆O₂.</p>	<p style="text-align: center;">% =</p>	<p>Quantitative content =</p> <p style="text-align: center;">$m_s =$ $V_{Na_2S_2O_3} =$</p>
<p style="text-align: center;">Conclusions</p>		

**PARACETAMOL
(PARACETAMOLUM)**



N-(4-Hydroxyphenyl)acetamide.
 $C_8H_9NO_2$

M.m. 151,2

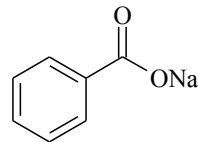
Paracetamol content: 99.0 per cent to 101.0 per cent (dried substance).

CHARACTERS		
Appearance: white, crystalline powder.		
Solubility: sparingly soluble in water, freely soluble in alcohol, very slightly soluble in methylene chloride		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
IDENTIFICATION		
A. Melting point (2.2.14): 168 °C to 172 °C.		
D. To 0.1 g add 1 ml of <i>hydrochloric acid R</i> , heat to boiling for 3 min, add 1 ml of <i>water R</i> and cool in an ice bath. No precipitate is formed. Add 0.05 ml of a 4.9 g/l solution of <i>potassium dichromate R</i> . A violet colour develops which does not change to red.		
E. It gives the reaction of acetyl (2.3.1). Heat over a naked flame. ACETYL In a test-tube about 180 mm long and 18 mm in external diameter, place about 15 mg of the substance to be examined, or the prescribed quantity, and 0.15 ml of <i>phosphoric acid R</i> . Close the tube with a stopper through which passes a small test-tube about 100 mm long and 10 mm in external diameter containing <i>water R</i> to act as a condenser. On the outside of the smaller tube, hang a drop of <i>lanthanum nitrate solution R</i> . Except for substances hydrolysable only with difficulty, place the apparatus in a water-bath for 5 min, then take out the smaller tube. Remove the drop and mix it with 0.05 ml of 0.01 M <i>iodine</i> on a tile. Add at the edge 0.05 ml of <i>dilute ammonia R2</i> . After 1 min to 2 min, a blue colour develops at the junction of the two		

<p>drops; the colour intensifies and persists for a short time. For <i>substances hydrolysable only with difficulty</i> heat the mixture slowly to boiling over an open flame and then proceed as prescribed above.</p>		
<p>TESTS Heavy metals (2.4.8): maximum 20 ppm. Dissolve 1.0 g in a mixture of 15 volumes of <i>water R</i> and 85 volumes of <i>acetone R</i> and dilute to 20 ml with the same mixture of solvents. 12 ml of the solution complies with limit test B. Prepare the standard using lead standard solution (1 ppm Pb) obtained by diluting <i>lead standard solution (100 ppm Pb) R</i> with a mixture of 15 volumes of <i>water R</i> and 85 volumes of <i>acetone R</i>.</p>		
<p>ASSAY Dissolve 0.300 g in a mixture of 10 ml of <i>water R</i> and 30 ml of <i>dilute sulphuric acid R</i>. Boil under a reflux condenser for 1 h, cool and dilute to 100.0 ml with <i>water R</i>. To 20.0 ml of the solution add 40 ml of <i>water R</i>, 40 g of ice, 15 ml of <i>dilute hydrochloric acid R</i> and 0.1 ml of <i>ferroin R</i>. Titrate with 0.1 M <i>cerium sulphate</i> until a greenish-yellow colour is obtained. Carry out a blank titration. 1 ml of 0.1 M <i>cerium sulphate</i> is equivalent to 7.56 mg of C₈H₉NO₂.</p>	<p style="text-align: center;">% =</p>	<p>Quantitative content =</p> $\frac{m_s}{V_{\text{Ce(SO}_4)_2}}$

<p><i>ferric chloride solution R1</i>. A dull-yellow precipitate, soluble in <i>ether R</i>, is formed.</p>		
<p>TESTS</p>		
<p>Solution S. Dissolve 5.0 g in <i>alcohol R</i> and dilute to 100 ml with the same solvent.</p>		
<p>Appearance of solution. Solution S is clear (2.2.1) and colourless (2.2.2, <i>Method II</i>).</p>		
<p>Carbonisable substances. Dissolve 0.5 g with shaking in 5 ml of <i>sulphuric acid R</i>. After 5 min, the solution is not more intensely coloured than reference solution Y₅ (2.2.2, <i>Method I</i>).</p>		
<p>Oxidisable substances. Dissolve 0.2 g in 10 ml of boiling <i>water R</i>. Cool, shake and filter. To the filtrate add 1 ml of <i>dilute sulphuric acid R</i> and 0.2 ml of 0.02 M <i>potassium permanganate</i>. After 5 min, the solution is still coloured pink.</p>		
<p>Heavy metals. (2.4.8). 12 ml of solution S complies with limit test B for heavy metals (10 ppm). Prepare the standard using a mixture of 5 ml of <i>lead standard solution (1 ppm Pb) R</i> and 5 ml of <i>alcohol R</i>.</p>		
<p>ASSAY Dissolve 0.200 g in 20 ml of <i>alcohol R</i> and titrate with 0.1 M <i>sodium hydroxide</i>, using 0.1 ml of <i>phenol red solution R</i> as indicator, until the colour changes from yellow to violet-red. 1 ml of 0.1 M <i>sodium hydroxide</i> is equivalent to 12.21 mg of C₇H₆O₂.</p>	<p style="text-align: center;">% =</p>	<p>Quantitative content =</p> <p style="text-align: center;">$m_s =$ $V_{NaOH} =$</p>
<p style="text-align: center;">Conclusions</p>		

SODIUM BENZOATE
(NATRII BENZOAS)



2-[[*(1R,S)*-1-Methyl-2-[3-(trifluoromethyl)phenyl]ethyl]amino]ethyl benzoate hydrochloride.
C₇H₅NaO₂ M.M. 144.1

Sodium benzoate contains not less than 99.0 per cent and not more than the equivalent of 100.5 per cent of sodium benzenecarboxylate, calculated with reference to the dried substance.

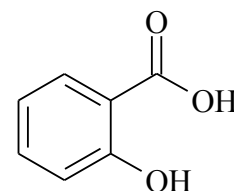
CHARACTERS		
A white, crystalline or granular powder or flakes, slightly hygroscopic, freely soluble in water, sparingly soluble in alcohol (90 per cent V/V).		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
IDENTIFICATION		
A. It gives reactions (b) and (c) of benzoates (2.3.1). BENZOATES b) Place 0.2 g of the substance to be examined, treated if necessary as prescribed, in a test-tube. Moisten with 0.2 ml to 0.3 ml of <i>sulphuric acid R</i> . Gently warm the bottom of the tube. A white sublimate is deposited on the inner wall of the tube. c) Dissolve 0.5 g of the substance to be examined in 10 ml of <i>water R</i> or use 10 ml of the prescribed solution. Add 0.5 ml of <i>hydrochloric acid R</i> . The precipitate obtained, after crystallisation from warm <i>water R</i> and drying <i>in vacuo</i> , has a melting point (2.2.14) of 120 °C to 124 °C.		
B. It gives reaction (a) of sodium (2.3.1). SODIUM a) Dissolve 0.1 g of the substance to be examined in 2 ml of <i>water R</i> or use 2 ml of the prescribed solution. Add 2 ml of a 150 g/l solution of <i>potassium carbonate R</i> and heat to boiling. No precipitate is formed. Add 4 ml of <i>potassium pyroantimonate solution R</i> and heat to boiling. Allow to cool in iced water and if necessary rub the inside of the test-tube with a glass rod. A dense white precipitate is formed.		
TESTS Solution S. Dissolve 10.0 g in <i>carbon dioxide-free water R</i> and dilute to 100 ml with the same solvent.		

<p>Appearance of solution. Solution S is clear (2.2.1) and not more intensely coloured than reference solution Y₆ (2.2.2, Method II)..</p>		
<p>Acidity or alkalinity. To 10 ml of solution S add 10 ml of <i>carbon dioxide-free water R</i> and 0.2 ml of <i>phenolphthalein solution R</i>. Not more than 0.2 ml of 0.1 M <i>sodium hydroxide</i> or 0.1 M <i>hydrochloric acid</i> is required to change the colour of the indicator.</p>		
<p>IRON Dissolve the prescribed quantity of the substance to be examined in <i>water R</i> and dilute to 10 ml with the same solvent or use 10 ml of the prescribed solution. Add 2 ml of a 200 g/l solution of <i>citric acid R</i> and 0.1 ml of <i>thioglycollic acid R</i>. Mix, make alkaline with <i>ammonia R</i> and dilute to 20 ml with <i>water R</i>. Prepare a standard in the same manner, using 10 ml of <i>iron standard solution (1 ppm Fe) R</i>. After 5 min, any pink colour in the test solution is not more intense than that in the standard</p>		
<p>SULPHATES <i>All solutions used for this test must be prepared with distilled water R.</i> Add ml of a 250 g/l solution of <i>barium chloride R</i> to 4.5 ml of <i>sulphate standard solution (10 ppm SO₄) RI</i>. Shake and allow to stand for 1 min. To 2.5 ml of this solution, add 15 ml of the solution to be examined and 0.5 ml of <i>acetic acid R</i>. Prepare a standard in the same manner using 15 ml of <i>sulphate standard solution (10 ppm SO₄) R</i> instead of the solution to be examined. After 5 min, any opalescence in the test solution is not more intense than that in the standard.</p>		
<p>ASSAY Dissolve 0.250 g in 20 ml of <i>anhydrous acetic acid R</i>, heating to 50 °C if necessary. Cool. Using 0.05 ml of <i>naphtholbenzein solution R</i> as indicator, titrate with 0.1 M <i>perchloric acid</i> until a green colour is obtained. 1 ml of 0.1 M <i>perchloric acid</i> is equivalent to 14.41 mg of C₇H₅NaO₂.</p>	<p style="text-align: center;">% = -----</p>	<p>Quantitative content =</p> <p style="text-align: center;">$\frac{m_s}{V_{HClO_4}}$</p>

<p style="text-align: right;"><i>N</i></p> <p>About 1,5 g of substance (accurate mass) dissolve in 20 ml of water in a flask with a stopper for 250 ml, add 45 ml of ether, 3-4 drops of the mixed indicator (1 ml of methyl orange and 1 ml of methylene blue) and titrate with 0,5 M hydrochloric acid solution till lilac colour in aqueous layer. Shake well the flask in the end of titration.</p> <p>1ml of 0,5 M hydrochloric acid is equivalent to 0,07205 g of $C_7H_5NaO_2$, which calculating on a dry substance must be not less than 99,0%.</p>	<p>% = -----</p>	<p>Quantitative content =</p> <p style="text-align: center;">$m_s =$ $V_{HCl} =$</p>
<p>Conclusions</p>		

SALICYLIC ACID

Acidum salicylicum



2-hydroxybenzenecarboxylic acid



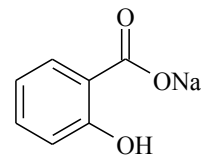
M_r 138.1

Salicylic acid contains not less than 99.0 per cent and not more than the equivalent of 100.5 per cent of 2-hydroxybenzenecarboxylic acid, calculated with reference to the dried substance.

<p>CHARACTERS</p> <p>A white, crystalline powder or white or colourless, acicular crystals, slightly soluble in water, freely soluble in alcohol and in ether, sparingly soluble in methylene chloride.</p>		
<p>Identification method</p> <p style="text-align: center;">1</p>	<p>Chemical equation</p> <p style="text-align: center;">2</p>	<p>Observations&Conclusions</p> <p style="text-align: center;">3</p>
<p>IDENTIFICATION</p> <p>Dissolve about 30 mg in 5 ml of 0.05 M sodium hydroxide, neutralise if necessary and dilute to 20 ml with water R. 1 ml of the solution gives reaction (a) of salicylates (2.3.1).</p> <p>SALICYLATES</p> <p>a) To 1 ml of the prescribed solution add</p>		

0.5 ml of <i>ferric chloride solution R1</i> . A violet colour is produced that persists after the addition of 0.1 ml of <i>acetic acid R</i> .		
TESTS Solution S. Dissolve 2.5 g in 50 ml of boiling <i>distilled water R</i> , cool and filter.		
Chlorides (2.4.4). 10 ml of solution S diluted to 15 ml with <i>water R</i> complies with the limit test for chlorides (100 ppm).		
Sulphates. Not more than 200 ppm. Dissolve 1.0 g in 5 ml of <i>dimethylformamide R</i> and add 4 ml of <i>water R</i> . Mix thoroughly. Add 0.2 ml of <i>dilute hydrochloric acid R</i> and 0.5 ml of a 25 per cent <i>m/m</i> solution of <i>barium chloride R</i> . After 15 min any opalescence in the solution is not more intense than that in a standard prepared as follows: to 2 ml of <i>sulphate standard solution (100 ppm SO₄) R</i> add 0.2 ml of <i>dilute hydrochloric acid R</i> , 0.5 ml of a 25 per cent <i>m/m</i> solution of <i>barium chloride R</i> , 3 ml of <i>water R</i> and 5 ml of <i>dimethylformamide R</i> .		
Heavy metals (2.4.8). Dissolve 2.0 g in 15 ml of <i>alcohol R</i> and add 5 ml of <i>water R</i> . 12 ml of the solution complies with limit test B for heavy metals (20 ppm). Prepare the standard using lead standard solution (2 ppm Pb) prepared by diluting <i>lead standard solution (100 ppm Pb) R</i> with a mixture of 5 volumes of <i>water R</i> and 15 volumes of <i>alcohol R</i> .		
ASSAY Dissolve 0.120 g in 30 ml of <i>alcohol R</i> and add 20 ml of <i>water R</i> . Titrate with 0.1 M <i>sodium hydroxide</i> , using 0.1 ml of <i>phenol red solution R</i> as indicator. 1 ml of 0.1 M <i>sodium hydroxide</i> is equivalent to 13.81 mg of C ₇ H ₆ O ₃ .	% = -----	Quantitative content = $\frac{m_s}{V_{NaOH}}$
Conclusions		

**SODIUM SALICYLATE
(NATRII SALICYLAS)**



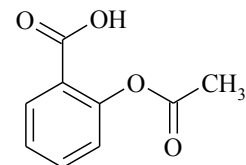
sodium 2-hydroxybenzenecarboxylate
 $C_7H_5NaO_3$

M.M. 160.1

Sodium salicylate contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of sodium 2-hydroxybenzenecarboxylate, calculated with reference to the dried substance.

CHARACTERS		
A white, crystalline powder or small, colourless crystals or shiny flakes, freely soluble in water, sparingly soluble in alcohol.		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
<p>IDENTIFICATION</p> <p>B. Solution S (see Tests) gives the reactions of salicylates (2.3.1).</p> <p>SALICYLATES</p> <p>a) To 1 ml of the prescribed solution add 0.5 ml of <i>ferric chloride solution R1</i>. A violet colour is produced that persists after the addition of 0.1 ml of <i>acetic acid R</i>.</p> <p>b) Dissolve 0.5 g of the substance to be examined in 10 ml of <i>water R</i> or use 10 ml of the prescribed solution. Add 0.5 ml of <i>hydrochloric acid R</i>. The precipitate obtained, after recrystallisation from hot <i>water R</i> and drying <i>in vacuo</i>, has a melting point (2.2.14) of 156 °C to 161 °C.</p>		
<p>C. It gives reaction (b) of sodium (2.3.1).</p> <p>SODIUM</p> <p>b) Dissolve a quantity of the substance to be examined equivalent to about 2 mg of sodium (Na^+) in 0.5 ml of <i>water R</i> or use 0.5 ml of the prescribed solution. Add 1.5 ml of <i>methoxyphenylacetic reagent R</i> and cool in ice-water for 30 min. A voluminous, white, crystalline precipitate is formed. Place in water at 20 °C and stir for 5 min. The precipitate does not disappear. Add 1 ml of <i>dilute ammonia R1</i>. The precipitate dissolves completely. Add 1 ml of <i>ammonium carbonate solution R</i>. No precipitate is formed.</p>		

**ACETYLSALYCILIC ACID
(ACIDUM ACETYLSALICYLICUM)**



2-(acetyloxy)benzoic acid
 $C_9H_8O_4$

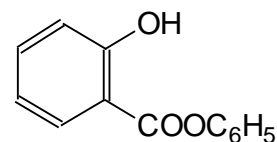
M.M. 180.2

Acetylsalicylic acid contains not less than 99.5 per cent and not more than the equivalent of 101.0 per cent of 2-(acetyloxy)benzoic acid, calculated with reference to the dried substance.

CHARACTERS		
A white, crystalline powder or colourless crystals, slightly soluble in water, freely soluble in alcohol, soluble in ether. It melts at about 143 °C (instantaneous method).		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
IDENTIFICATION		
B. To 0.2 g add 4 ml of <i>dilute sodium hydroxide solution R</i> and boil for 3 min. Cool and add 5 ml of <i>dilute sulphuric acid R</i> . A crystalline precipitate is formed. Filter, wash the precipitate and dry at 100 °C to 105 °C. The melting point (2.2.14) is 156 °C to 161 °C.		
C. In a test tube mix 0.1 g with 0.5 g of <i>calcium hydroxide R</i> . Heat the mixture and expose to the fumes produced a piece of filter paper impregnated with 0.05 ml of <i>nitrobenzaldehyde solution R</i> . A greenish-blue or greenish-yellow colour develops on the paper. Moisten the paper with <i>dilute hydrochloric acid R</i> . The colour becomes blue.		
D. Dissolve with heating about 20 mg of the precipitate obtained in identification test B in 10 ml of <i>water R</i> and cool. The solution gives reaction (a) of salicylates (2.3.1). SALICYLATES a) To 1 ml of the prescribed solution add 0.5 ml of <i>ferric chloride solution R1</i> . A violet colour is produced that persists after the addition of 0.1 ml of <i>acetic acid R</i> .		

TESTS		
Appearance of solution (Dissolve 1.0 g in 9 ml of <i>alcohol R</i> . The solution is clear (2.2.1) and colourless (2.2.2, <i>Method II</i>).		
Heavy metals. (2.4.8). Dissolve 1.0 g in 12 ml of <i>acetone R</i> and dilute to 20 ml with <i>water R</i> . 12 ml of this solution complies with limit test B for heavy metals (20 ppm). Prepare the standard using lead standard solution (1 ppm Pb) obtained by diluting <i>lead standard solution (100 ppm Pb) R</i> with a mixture of 6 volumes of <i>water R</i> and 9 volumes of <i>acetone R</i> .		
ASSAY In a flask with a ground-glass stopper, dissolve 1.000 g in 10 ml of <i>alcohol R</i> . Add 50.0 ml of 0.5 M <i>sodium hydroxide</i> . Close the flask and allow to stand for 1 h. Using 0.2 ml of <i>phenolphthalein solution R</i> as indicator, titrate with 0.5 M <i>hydrochloric acid</i> . Carry out a blank titration. 1 ml of 0.5 M <i>sodium hydroxide</i> is equivalent to 45.04 mg of C ₉ H ₈ O ₄ .	% = -----	Quantitative content = m_s= V_{HCl}=
Conclusions		

PHENYLII SALICYLAS
PHENYLIUM SALICYLICUM



2-Hydroxyethyl 2-hydroxybenzoate.
C₁₃H₁₀O₃

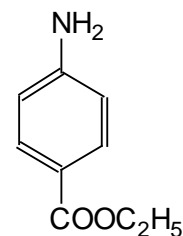
M.M. 214,22

CHARACTERS		
A white, crystalline powder or colourless crystals, very slightly soluble in water, freely soluble in alcohol and in ether.		
Identification method	Chemical equation	Observations & Conclusions
1	2	3
IDENTIFICATION 0,02 g of preparation dissolve in 2 ml of alcohol and add 1 drop of ferric chloride solution - a violet colour is produced		

0,02 g of preparation add 3-4 drops of conc. sulphuric acid and 1-2 drops of water. The smell of phenol is produced. Then add 1-2 drops of formaldehyde solution – pink colour appears.		
Conclusions		

Theme: MEDICINAL SUBSTANCES WHICH ARE DERIVATIVES OF AROMATIC AMINOACIDS

BENZOCAINE
Benzocainum



Ethyl 4-aminobenzoate



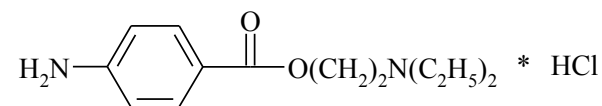
M_r 165.2

Benzocaine contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of ethyl 4-aminobenzoate, calculated with reference to the dried substance.

CHARACTERS		
A white, crystalline powder or colourless crystals, very slightly soluble in water, freely soluble in alcohol and in ether.		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
IDENTIFICATION		
C. To about 50 mg in a test-tube add 0.2 ml of a 500 g/l solution of <i>chromium trioxide R</i> . Cover the mouth of the tube with a piece of filter paper moistened with a freshly prepared mixture of equal volumes of a 50 g/l solution of <i>sodium nitroprusside R</i> and a 200 g/l solution of <i>piperazine hydrate R</i> . Boil gently for at least 30 s. A blue colour develops on the filter paper.		

<p>TESTS</p> <p>Appearance of solution. Dissolve 1.0 g in <i>alcohol R</i> and dilute to 20 ml with the same solvent. The solution is clear (2.2.1) and colourless (2.2.2, <i>Method II</i>).</p> <p>Acidity or alkalinity. Dissolve 0.5 g in 10 ml of <i>alcohol R</i> previously neutralised to 0.05 ml of <i>phenolphthalein solution R</i>. Add 10 ml of <i>carbon dioxide-free water R</i>. The solution remains colourless and not more than 0.5 ml of 0.01 M <i>sodium hydroxide</i> is required to change the colour of the indicator.</p>		
<p>ASSAY</p> <p>Dissolve 0.400 g in a mixture of 25 ml of <i>hydrochloric acid R</i> and 50 ml of <i>water R</i>. Carry out the determination of primary aromatic amino-nitrogen (2.5.8).</p> <p>1 ml of 0.1 M <i>sodium nitrite</i> is equivalent to 16.52 mg of $C_9H_{11}NO_2$.</p> <p>2.5.8. DETERMINATION OF PRIMARY AROMATIC AMINO-NITROGEN</p> <p>Dissolve the prescribed quantity of the substance to be examined in 50 ml of <i>dilute hydrochloric acid R</i> or in another prescribed solvent and add 3 g of <i>potassium bromide R</i>. Cool in ice-water and titrate by slowly adding 0.1 M <i>sodium nitrite</i> with constant stirring.</p> <p>Determine the end-point electrometrically or by the use of the prescribed indicator.</p>	<p style="text-align: center;">% = -----</p>	<p>Quantitative content =</p> <p style="text-align: center;">$m_s =$ $V_{NaNO_2} =$</p>
<p style="text-align: center;">Conclusions</p>		

PROCAINE HYDROCHLORIDE
(*PROCAINI HYDROCHLORIDUM*)



2-(diethylamino)ethyl 4-aminobenzoate hydrochloride
C₁₃H₂₁ClN₂O₂

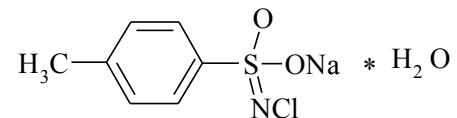
M.M. 272,8

Procaine hydrochloride contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of 2-(diethylamino)ethyl 4-aminobenzoate hydrochloride, calculated with reference to the dried substance.

CHARACTERS		
A white, crystalline powder or colourless crystals, very soluble in water, soluble in alcohol, practically insoluble in ether.		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
IDENTIFICATION		
C. To about 5 mg add 0.5 ml of fuming nitric acid R. Evaporate to dryness on a water-bath, allow to cool and dissolve the residue in 5 ml of acetone R. Add 1 ml of 0.1 M alcoholic potassium hydroxide. Only a brownish-red colour develops.		
D. To 0.2 ml of solution S (see Tests) add 2 ml of water R and 0.5 ml of dilute sulphuric acid R and shake. Add 1 ml of a 1 g/l solution of potassium permanganate R. The colour is immediately discharged.		
E. It gives reaction (a) of chlorides (2.3.1). CHLORIDES a) Dissolve in 2 ml of water R a quantity of the substance to be examined equivalent to about 2 mg of chloride (Cl ⁻) or use 2 ml of the prescribed solution. Acidify with dilute nitric acid R and add 0.4 ml of silver nitrate solution R1. Shake and allow to stand. A curdled, white precipitate is formed. Centrifuge and wash the precipitate with three quantities, each of 1 ml, of water R. Carry out this operation rapidly in subdued light, disregarding the fact that the supernatant solution may not become perfectly clear. Suspend the precipitate in 2 ml of water R and add 1.5 ml of ammonia R. The precipitate dissolves easily with the possible exception of a few large particles which dissolve slowly.		

<p>F. Dilute 1 ml of solution S to 100 ml with <i>water R</i>. 2 ml of this solution gives the reaction of primary aromatic amines (2.3.1).</p> <p>AMINES, PRIMARY AROMATIC</p> <p>Acidify the prescribed solution with <i>dilute hydrochloric acid R</i> and add 0.2 ml of <i>sodium nitrite solution R</i>. After 1 min to 2 min, add 1 ml of <i>β-naphthol solution R</i>. An intense orange or red colour and usually a precipitate of the same colour are produced</p>		
<p>TESTS</p> <p>Solution S. Dissolve 2.5 g in <i>carbon dioxide-free water R</i> and dilute to 50 ml with the same solvent.</p> <p>Appearance of solution. Solution S is clear (2.2.1) and colourless (2.2.2, <i>Method II</i>).</p> <p>pH. (2.2.3). Dilute 4 ml of solution S to 10 ml with <i>carbon dioxide-free water R</i>. The pH of the solution is 5.0 to 6.5.</p>		
<p>ASSAY</p> <p>Dissolve 0.400 g in 50 ml of <i>dilute hydrochloric acid R</i>. Carry out the determination of primary aromatic amino nitrogen (2.5.8).</p> <p>1 ml of 0.1 M <i>sodium nitrite</i> is equivalent to 27.28 mg of C₁₃H₂₁ClN₂O₂.</p> <p>2.5.8. DETERMINATION OF PRIMARY AROMATIC AMINO-NITROGEN</p> <p>Dissolve the prescribed quantity of the substance to be examined in 50 ml of <i>dilute hydrochloric acid R</i> or in another prescribed solvent and add 3 g of <i>potassium bromide R</i>. Cool in ice-water and titrate by slowly adding 0.1 M <i>sodium nitrite</i> with constant stirring. Determine the end-point electrometrically or by the use of the prescribed indicator.</p>	<p style="text-align: center;">% = -----</p>	<p>Quantitative content =</p> <p style="text-align: center;">$m_s =$ $V_{NaNO_2} =$</p>
<p style="text-align: center;">Conclusions</p>		

CHLORAMINE
(Chloraminum)



sodium *N*-chloro-4-methylbenzene-sulphonimidate trihydrate.

$C_7H_7ClNaO_2S \cdot 3H_2O$

M.M. 281,7

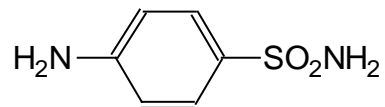
Chloramine contains not less than 98.0 per cent and not more than the equivalent of 103.0 per cent of sodium *N*-chloro-4-methylbenzene-sulphonimidate trihydrate.

CHARACTERS		
A white or slightly yellow, crystalline powder, freely soluble in water, soluble in alcohol, practically insoluble in ether.		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
IDENTIFICATION		
A. Solution S (see Tests) turns <i>red litmus paper R</i> blue and then bleaches it.		
B. To 10 ml of solution S add 10 ml of <i>dilute hydrogen peroxide solution R</i> . A white precipitate is formed which dissolves on heating. Filter the hot solution and allow to cool. White crystals are formed which, when washed and dried at 100 °C to 105 °C, melt (2.2.14) at 137 °C to 140 °C.		
C. Ignite 1 g (cautiously, because of the risk of deflagration). Dissolve the residue in 10 ml of <i>water R</i> . The solution gives reaction (a) of chlorides (2.3.1). a) Dissolve in 2 ml of <i>water R</i> a quantity of the substance to be examined equivalent to about 2 mg of chloride (Cl ⁻) or use 2 ml of the prescribed solution. Acidify with <i>dilute nitric acid R</i> and add 0.4 ml of <i>silver nitrate solution R1</i> . Shake and allow to stand. A curdled, white precipitate is formed. Centrifuge and wash the precipitate with three quantities, each of 1 ml, of <i>water R</i> . Carry out this operation rapidly in subdued light, disregarding the fact that the supernatant solution may not become perfectly clear. Suspend the precipitate in 2 ml of <i>water R</i> and add 1.5 ml of <i>ammonia R</i> . The precipitate dissolves easily with the possible exception of a few large particles which dissolve slowly.		

<p>D. The solution prepared for identification test C gives reaction (a) of sulphates (2.3.1). SULPHATES a) Dissolve about 45 mg of the substance to be examined in 5 ml of <i>water R</i> or use 5 ml of the prescribed solution. Add 1 ml of <i>dilute hydrochloric acid R</i> and 1 ml of <i>barium chloride solution RI</i>. A white precipitate is formed.</p>		
<p>E. The solution prepared for identification test C gives reaction (b) of sodium (2.3.1). SODIUM b) Dissolve a quantity of the substance to be examined equivalent to about 2 mg of sodium (Na^+) in 0.5 ml of <i>water R</i> or use 0.5 ml of the prescribed solution. Add 1.5 ml of <i>methoxyphenylacetic reagent R</i> and cool in ice-water for 30 min. A voluminous, white, crystalline precipitate is formed. Place in water at 20 °C and stir for 5 min. The precipitate does not disappear. Add 1 ml of <i>dilute ammonia RI</i>. The precipitate dissolves completely. Add 1 ml of <i>ammonium carbonate solution R</i>. No precipitate is formed.</p>		
<p>TESTS Solution S. Dissolve 1.0 g in <i>carbon dioxide-free water R</i> and dilute to 20 ml with the same solvent.</p>		
<p>Appearance of solution. Solution S is not more opalescent than reference suspension II (2.2.1) and is colourless (2.2.2, <i>Method II</i>).</p>		
<p>pH. (2.2.3). The pH of solution S is 8.0 to 10.0.</p>		
<p>ASSAY Dissolve 0.125 g in 100 ml of <i>water R</i> in a ground-glass-stoppered flask. Add 1 g of <i>potassium iodide R</i> and 5 ml of <i>dilute sulphuric acid R</i>. Allow to stand for 3 min. Titrate with 0.1 M <i>sodium thiosulphate</i>, using 1 ml of <i>starch solution R</i> as indicator. 1 ml of 0.1 M <i>sodium thiosulphate</i> is equivalent to 14.08 mg of $\text{C}_7\text{H}_7\text{ClNNaO}_2\text{S}\cdot 3\text{H}_2\text{O}$.</p>	<p style="text-align: center;">% =</p>	<p>Quantitative content =</p> <p style="text-align: center;">$m_s =$ $V_{\text{Na}_2\text{S}_2\text{O}_3} =$</p>
<p style="text-align: center;">Conclusions</p>		

Theme: MEDICINAL SUBSTANCES WHICH ARE DERIVATIVES OF AROMATIC SULPHOACIDS

**SULFANILAMIDE
(SULFANILAMIDUM)**



4-aminobenzenesulphonamide



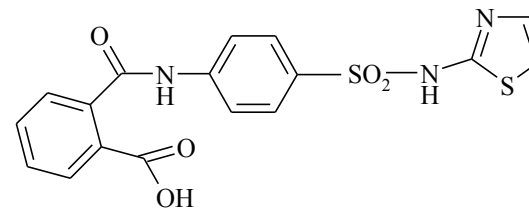
M.M 172.2

Sulfanilamide contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of 4-aminobenzenesulphonamide, calculated with reference to the dried substance.

CHARACTERS		
White or yellowish-white crystals or fine powder, slightly soluble in water, freely soluble in acetone, sparingly soluble in alcohol, practically insoluble in methylene chloride. It dissolves in solutions of alkali hydroxides and in dilute mineral acids.		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
IDENTIFICATION		
A. Melting point (2.2.14): 164.5 °C to 166.0 °C.		
D. Dissolve about 5 mg in 10 ml of 1 M hydrochloric acid. Dilute 1 ml of the solution to 10 ml with water R. The solution, without further acidification, gives the reaction of primary aromatic amines (2.3.1).		
AMINES, PRIMARY AROMATIC		
Acidify the prescribed solution with dilute hydrochloric acid R and add 0.2 ml of sodium nitrite solution R. After 1 min to 2 min, add 1 ml of β-naphthol solution R. An intense orange or red colour and usually a precipitate of the same colour are produced.		
_____N		
0,1 g of substance heat in a dry test-tube on a flame; the product of melting is of violet-blue colour and the smell of ammonia and aniline appear.		

TESTS		
Solution S. To 2.5 g add 50 ml of <i>carbon dioxide-free water R</i> . Heat at about 70 °C for about 5 min. Cool in iced water for about 15 min and filter.		
Acidity. To 20 ml of solution S add 0.1 ml of <i>bromothymol blue solution R1</i> . Not more than 0.2 ml of 0.1 M sodium hydroxide is required to change the colour of the indicator.		
ASSAY Carry out the determination of primary aromatic amino-nitrogen (2.5.8), using 0.140 g and determining the end-point electrometrically. 1 ml of 0.1 M sodium nitrite is equivalent to 17.22 mg of C ₆ H ₈ N ₂ O ₂ S. 2.5.8. DETERMINATION OF PRIMARY AROMATIC AMINO-NITROGEN Dissolve the prescribed quantity of the substance to be examined in 50 ml of <i>dilute hydrochloric acid R</i> or in another prescribed solvent and add 3 g of <i>potassium bromide R</i> . Cool in ice-water and titrate by slowly adding 0.1 M sodium nitrite with constant stirring. Determine the end-point electrometrically or by the use of the prescribed indicator.	$\% = \text{-----}$	Quantitative content = $m_s =$ $V_{NaNO_2} =$
Conclusions		

PHTHALYLSULFATHIAZOLE
Phthalylsulfathiazolum
(PHTHALAZOLUM)



2-[[4-(thiazol-2-ylsulphamoyl)phenyl]carbamoyl]benzoic acid

$C_{17}H_{13}N_3O_5S_2$

M_M 403,4

Phthalylsulfathiazole contains not less than 98.5 per cent and not more than the equivalent of 101.5 per cent of 2-[[4-(thiazol-2-ylsulphamoyl)phenyl]carbamoyl]benzoic acid, calculated with reference to the dried substance.

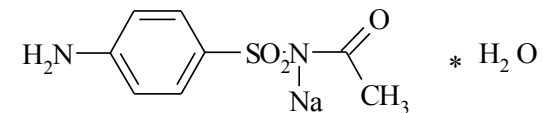
CHARACTERS

A white or yellowish-white, crystalline powder, practically insoluble in water and in ether, freely soluble in dimethylformamide, slightly soluble in acetone and in alcohol.

Identification method	Chemical equation	Observations&Conclusions
1	2	3
<p>IDENTIFICATION D. To 0.1 g add 0.5 g of <i>resorcinol R</i> and 0.3 ml of <i>sulphuric acid R</i> and heat on a water-bath until a homogeneous mixture is obtained. Allow to cool. Add 5 ml of <i>dilute sodium hydroxide solution R</i>. Dilute 0.1 ml of this brownish-red mixture to 25 ml with <i>water R</i>. An intense green fluorescence appears which disappears on acidification.</p>		

<p>E. Dissolve about 10 mg of the crystals obtained in identification test B in 200 ml of 0.1 M hydrochloric acid. 2 ml of the solution gives the reaction of primary aromatic amines (2.3.1) with formation of an orange precipitate.</p> <p>AMINES, PRIMARY AROMATIC Acidify the prescribed solution with dilute hydrochloric acid R and add 0.2 ml of sodium nitrite solution R. After 1 min to 2 min, add 1 ml of β-naphthol solution R. An intense orange or red colour and usually a precipitate of the same colour are produced.</p>		
<p>TESTS Acidity. To 2.0 g add 20 ml of water R, shake continuously for 30 min and filter. To 10 ml of the filtrate add 0.1 ml of phenolphthalein solution R. Not more than 0.2 ml of 0.1 M sodium hydroxide is required to change the colour of the indicator.</p>		
<p>ASSAY Dissolve 0.300 g in 40 ml of dimethylformamide R. Titrate with 0.1 M sodium hydroxide until the colour becomes blue using 0.2 ml of thymolphthalein solution R as indicator. Carry out a blank titration. 1 ml of 0.1 M sodium hydroxide is equivalent to 20.17 mg of C₁₇H₁₃N₃O₅S₂.</p>	<p style="text-align: center;">% =</p>	<p>Quantitative content =</p> $\frac{m_s}{V_{\text{NaOH}}}$
<p>Conclusions</p>		

SULFACETAMIDE SODIUM
(*SULFACETAMIDUM NATRICUM*)



Sodium derivative of *N*-[(4-aminophenyl)sulphonyl]acetamide

$C_8H_9N_2NaO_3S \cdot H_2O$

M_M 254,24

Sulfacetamide sodium contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of the sodium derivative of *N*-[(4-aminophenyl)sulphonyl]acetamide, calculated with reference to the anhydrous substance.

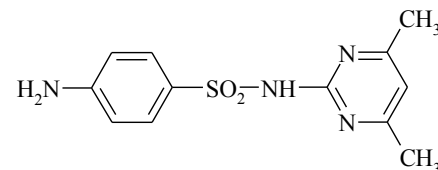
CHARACTERS		
A white or yellowish-white, crystalline powder, freely soluble in water, slightly soluble in ethanol, practically insoluble in ether.		
Identification method	Chemical equation	Observations & Conclusions
1	2	3
IDENTIFICATION C. Dissolve 1 g in 10 ml of <i>water R</i> , add 6 ml of <i>dilute acetic acid R</i> and filter. The precipitate, washed with a small quantity of <i>water R</i> and dried at 100 °C to 105 °C for 4 h, melts (2.2.14) at 181 °C to 185 °C.		
D. Dissolve 0.1 g of the precipitate obtained in identification test C in 5 ml of <i>alcohol R</i> . Add 0.2 ml of <i>sulphuric acid R</i> and heat. The odour of ethyl acetate is perceptible.		
E. Dissolve about 1 mg of the precipitate obtained in identification test C, with heating, in 1 ml of <i>water R</i> . The solution gives the reaction of primary aromatic amines (2.3.1) with formation of an orange-red precipitate. AMINES, PRIMARY AROMATIC Acidify the prescribed solution with <i>dilute hydrochloric acid R</i> and add 0.2 ml of <i>sodium nitrite solution R</i> . After 1 min to 2 min, add 1 ml of <i>β-naphthol solution R</i> . An intense orange or red colour and usually a precipitate of the same colour are produced.		

<p>F. Solution S (see Tests) gives the reactions of sodium (2.3.1).</p> <p>SODIUM</p> <p>a) Dissolve 0.1 g of the substance to be examined in 2 ml of <i>water R</i> or use 2 ml of the prescribed solution. Add 2 ml of a 150 g/l solution of <i>potassium carbonate R</i> and heat to boiling. No precipitate is formed. Add 4 ml of <i>potassium pyroantimonate solution R</i> and heat to boiling. Allow to cool in iced water and if necessary rub the inside of the test-tube with a glass rod. A dense white precipitate is formed.</p> <p>b) Dissolve a quantity of the substance to be examined equivalent to about 2 mg of sodium (Na^+) in 0.5 ml of <i>water R</i> or use 0.5 ml of the prescribed solution. Add 1.5 ml of <i>methoxyphenylacetic reagent R</i> and cool in ice-water for 30 min. A voluminous, white, crystalline precipitate is formed. Place in water at 20 °C and stir for 5 min. The precipitate does not disappear. Add 1 ml of <i>dilute ammonia R1</i>. The precipitate dissolves completely. Add 1 ml of <i>ammonium carbonate solution R</i>. No precipitate is formed.</p>		
<p>_____N</p> <p>0,1 g of substance dissolve in 3 ml of water and add 1 ml of copper sulphate solution; bluish-green precipitate appears.</p>		
<p>TESTS</p> <p>Solution S. Dissolve 1.25 g in <i>carbon dioxide-free water R</i> and dilute to 25 ml with the same solvent.</p>		
<p>pH (2.2.3). The pH of solution S is 8.0 to 9.5.</p>		
<p>Sulphates (2.4.13). Dissolve 2.5 g in <i>distilled water R</i> and dilute to 25 ml with the same solvent. Add 25 ml of <i>dilute acetic acid R</i>, shake for 30 min and filter. 15 ml of the filtrate complies with the limit test for sulphates (200 ppm).</p>		

<p>Heavy metals (2.4.8). 12 ml of the filtrate obtained in the test for sulphates complies with limit test A for heavy metals (20 ppm). Prepare the standard using <i>lead standard solution (1 ppm Pb) R</i>.</p>		
<p>ASSAY Dissolve 0.500 g in a mixture of 50 ml of <i>water R</i> and 20 ml of <i>dilute hydrochloric acid R</i>. Cool the solution in iced water and carry out the determination of primary aromatic amino-nitrogen (2.5.8), determining the end-point electrometrically. 1 ml of 0.1 M <i>sodium nitrite</i> is equivalent to 23.62 mg of C₈H₉N₂NaO₃S.</p> <p>2.5.8. DETERMINATION OF PRIMARY AROMATIC AMINO-NITROGEN Dissolve the prescribed quantity of the substance to be examined in 50 ml of <i>dilute hydrochloric acid R</i> or in another prescribed solvent and add 3 g of <i>potassium bromide R</i>. Cool in ice-water and titrate by slowly adding 0.1 M <i>sodium nitrite</i> with constant stirring.</p>	<p>% =</p>	<p>Quantitative content =</p> <p>$m_s =$</p> <p>$V_{NaNO_2} =$</p>
<p>Conclusions</p>		

Sulfadimidine

(Sulfadimezinum)



4-amino-*N*-(4,6-dimethylpyrimidin-2-yl)

C₁₂H₁₄N₄O₂S

M_M 278.3

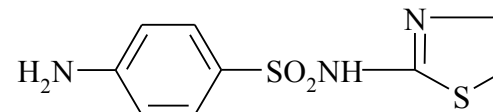
Sulfadimidine contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of 4-amino-*N*-(4,6-dimethylpyrimidin-2-yl)benzenesulphonamide, calculated with reference to the dried substance.

<p>CHARACTERS White or almost white powder or crystals, very slightly soluble in water and in ether, soluble in acetone, slightly soluble in alcohol. It dissolves in solutions of alkali hydroxides and in dilute mineral acids.</p>		
<p>Identification method</p>	<p>Chemical equation</p>	<p>Observations&Conclusions</p>
<p>1</p>	<p>2</p>	<p>3</p>

<p>IDENTIFICATION</p> <p>D. Dissolve about 5 mg in 10 ml of 1 M hydrochloric acid. Dilute 1 ml of the solution to 10 ml with water R. The solution, without further acidification, gives the reaction of primary aromatic amines (2.3.1).</p> <p>AMINES, PRIMARY AROMATIC</p> <p>Acidify the prescribed solution with dilute hydrochloric acid R and add 0.2 ml of sodium nitrite solution R. After 1 min to 2 min, add 1 ml of β-naphthol solution R. An intense orange or red colour and usually a precipitate of the same colour are produced.</p>		
<p style="text-align: center;">N</p> <p>Dissolve about 100 mg in a mixture of 10 ml of water R and 2 ml of 0.1 M sodium hydroxide, shake for 2 min and filter; to the filter add 0.5 ml of copper sulphate solution R. A yellow-green precipitate is formed in time getting brown.</p>		
<p>To 0.1 g of substance add 1 ml of water, 6 drops of sodium nitroprusside solution R. At mixing violet colour appears.</p>		
<p>TESTS</p> <p>Acidity. To 1.25 g, finely powdered, add 25 ml of carbon dioxide-free water R. Heat at about 70 °C for 5 min. Cool in iced water for about 15 min and filter. To 20 ml of the filtrate add 0.1 ml of bromothymol blue solution R1. Not more than 0.2 ml of 0.1 M sodium hydroxide is required to change the colour of the indicator.</p>		
<p>Heavy metals (2.4.8). 1.0 g complies with limit test D for heavy metals (20 ppm). Prepare the standard using 2 ml of lead standard solution (10 ppm Pb) R.</p>		

<p>ASSAY Dissolve 0.250 g in a mixture of 20 ml of <i>dilute hydrochloric acid R</i> and 50 ml of <i>water R</i>. Cool the solution in iced water. Carry out the determination of primary aromatic amino- nitrogen (2.5.8), determining the end-point electrometrically. 1 ml of 0.1 M <i>sodium nitrite</i> is equivalent to 27.83 mg of C₁₂H₁₄N₄O₂S.</p> <p>DETERMINATION OF PRIMARY AROMATIC AMINO-NITROGEN Dissolve the prescribed quantity of the substance to be examined in 50 ml of <i>dilute hydrochloric acid R</i> or in another prescribed solvent and add 3 g of <i>potassium bromide R</i>. Cool in ice-water and titrate by slowly adding 0.1 M <i>sodium nitrite</i> with constant stirring. Determine the end-point electrometrically or by the use of the prescribed indicator.</p>	<p style="text-align: center;">% = -----</p>	<p>Quantitative content =</p> <p style="text-align: center;">$m_s =$ $V_{NaNO_2} =$</p>
<p style="text-align: center;">Conclusions</p>		

Sulfathiazole
(Norsulfazolum)



4-amino-*N*-(thiazol-2-yl)benzenesulphonamide
C₉H₉N₃O₂S₂

M_r 255,32

Sulfathiazole contains not less than 99.0 per cent and not more than the equivalent of 101.0 per cent of 4-amino-*N*-(thiazol-2-yl)benzenesulphonamide, calculated with reference to the dried substance.

CHARACTERS		
A white or slightly yellowish, crystalline powder, practically insoluble in water, slightly soluble in alcohol, practically insoluble in ether and in methylene chloride. It dissolves in dilute solutions of alkali hydroxides and in dilute mineral acids.		
Identification method	Chemical equation	Observations&Conclusions
1	2	3
IDENTIFICATION		
A. Melting point (2.2.14): 200 °C to 203 °C. Melting may occur at about 175 °C, followed by solidification and a second melting between 200 °C and 203 °C.		
D. Dissolve about 10 mg in a mixture of 10 ml of <i>water R</i> and 2 ml of 0.1 M <i>sodium hydroxide</i> and add 0.5 ml of <i>copper sulphate solution R</i> . A greyish-blue or purple precipitate is formed.		
E. Dissolve about 5 mg in 10 ml of 1 M <i>hydrochloric acid</i> . Dilute 1 ml of the solution to 10 ml with <i>water R</i> . The solution, without further addition of acid, gives the reaction of primary aromatic amines (2.3.1).		
AMINES, PRIMARY AROMATIC Acidify the prescribed solution with <i>dilute hydrochloric acid R</i> and add 0.2 ml of <i>sodium nitrite solution R</i> . After 1 min to 2 min, add 1 ml of <i>β-naphthol solution R</i> . An intense orange or red colour and usually a precipitate of the same colour are produced.		

<p style="text-align: center;"><i>N</i></p> <p>0,05 g of substance heat in a dry test-tube on an open flame; the product of melting of brown colour appears.</p>		
<p>TESTS Acidity. To 1.0 g add 50 ml of <i>carbon dioxide-free water R</i>. Heat to 70 °C for 5 min. Cool rapidly to 20 °C and filter. To 25 ml of the filtrate add 0.1 ml of <i>bromothymol blue solution R1</i>. Not more than 0.1 ml of 0.1 M <i>sodium hydroxide</i> is required to change the colour of the indicator.</p>		
<p>Heavy metals (2.4.8). 1.0 g complies with limit test C for heavy metals (20 ppm). Prepare the standard using 2 ml of <i>lead standard solution (10 ppm Pb) R</i>.</p>		
<p>ASSAY Carry out the determination of primary aromatic amino- nitrogen (2.5.8), using 0.200 g, determining the end-point electrometrically. 1 ml of 0.1 M <i>sodium nitrite</i> is equivalent to 25.53 mg of C₉H₉N₃O₂S₂.</p> <p>DETERMINATION OF PRIMARY AROMATIC AMINO-NITROGEN Dissolve the prescribed quantity of the substance to be examined in 50 ml of <i>dilute hydrochloric acid R</i> or in another prescribed solvent and add 3 g of <i>potassium bromide R</i>. Cool in ice-water and titrate by slowly adding 0.1 M <i>sodium nitrite</i> with constant stirring. Determine the end-point electrometrically or by the use of the prescribed indicator.</p>	<p>% =</p>	<p>Quantitative content =</p> <p style="text-align: center;">$m_s =$ $V_{NaNO_2} =$</p>
<p>Conclusions</p>		

Theme: Analysis of medicinal forms

Rp.:

Identification method	Chemical equation		Observations&Conclusions
1	2		3
Organoleptic control (test of an appearance): color, odour, transparency, mechanical impurity absence.			
Physical control: general volume test of dosage form.		Absolute deviation = Relative deviation = Acceptable deviation (Order Ukr.№ 626, suppl. №5) =	
Chemical control IDENTIFICATION:			

Assay:	X, g = -----		
	Absolute deviation =	Relative deviation =	Acceptable deviation (Order Ukr.№ 626, suppl. №) =
Conclusions			

Rp.:

Identification method	Chemical equation		Observations&Conclusions
1	2		3
Organoleptic control (test of an appearance): color, odour, transparency, mechanical impurity absence.			
Physical control: general volume test of dosage form.		Absolute deviation = Relative deviation = Acceptable deviation (Order Ukr.№ 626, suppl. №5) =	
Chemical control IDENTIFICATION:			

Assay:	$X, g = \text{-----}$		
	Absolute deviation =	Relative deviation =	Acceptable deviation (Order Ukr.№ 626, suppl. №) =
Conclusions			