Ministry of Health of Ukraine
National Pirogov Memorial Medical University, Vinnytsya

Department of Pharmaceutical Chemistry

«AGREED»
with the Methodical Council
of the Pharmaceutical Faculty
Minutes № 2
Dated «23» 12 2024

Head of the Methodical Council
of the Pharmaceutical Faculty

_____ assoc. prof. of HEI Tetyana YUSHCHENKO

«APPROVED»
by the Academic Council of Stomatological
and Pharmaceutical Faculty
Minutes № 2
Dated «24» 12 2024

Head of the Academic Council of
Stomatological and Pharmaceutical Faculty

_____ professor of HEI Serhiy POLISHCHUK

Instruction for the Station
Objective Structured Practical Examination (OSPE)

Station name	***Station №5. Quality control of Medicines.***
Subject	Pharmaceutical chemistry
Speciality	226 «Pharmacy, industrial pharmacy»
Educational qualification	Master of pharmacy
Professional qualification	Pharmacist
Course	V
Form of study	Full-time

Vinnytsya 2024

Instructions to the station № 5
«Quality control of medicines»

Tasks:
1. Identify the substance by cations, anions and functional groups and indicate their analytical effects;
2. Specify the name of method of quantitative determination (assay), titrant, way of fixing the equivalence point and color transition, calculate the quantitative content in the substance and draw a conclusion about the quality.

Station equipment:
1. Scenario of a practical situation.
2. Medicinal substances, chemical utensils and reagents.
3. A4 paper.
4. Pen.
5. Calculator.

In the case of **distance form of studying** (martial law, emergency situations or a state of emergency (special period)) **the procedure for conducting an objective structured practical examination (OSPI)** is governed by the Regulation on the introduction of elements of distance learning in VNMU named after M. I. Pirogov and will take place on the **Microsoft Teams platform.**

Equipment for remote form of OSPI: practical situation, method of quantitative determination, calculator.

On the day of the exam, the secretary of the State Examination Commission joins the meeting of the examiner of the student, the group, which passes the exam according to the schedule. At the station, the student must greet and introduce himself, present a document (passport) proving his identity to the teacher.

The student receives a practical situation, which provides to test the ability of higher education to organize and carry out the quality control of medicines of different pharmacological groups (qualitative analysis of cations and anions; elemental analysis and analysis of organic compounds by functional groups; chemical titrimetric methods of analysis, etc.) in accordance with the requirements of current State Pharmacopoeia of Ukraine and methods of quality control.

Duration of work at the station: 8 minutes. After the end of the stay at the station, the examiner does not accept the answer. Note that the teacher is an observer of your actions and does not provide instructions, comment or question.

Requirements for passing the station:
- use a computer or laptop during the response, use of calculator;
- the answer is accepted under the condition of the included camera, where the student who passes the exam is clearly visible, and the included microphone with a clear sound;
- the calculations are sent to the chat;
- video is recorded while working at the station.

It is forbidden to use a mobile phone and other electronic gadgets, to transmit, copy and take out any information related to the exam.

Part of OSPI on pharmaceutical chemistry includes station №5 "Quality control of medicines"

Practical situations on analysis of quality of medicines that affect on central nervous, cardiovascular, excretory systems and system of blood coagulation, antimicrobic medicines and medicines that affect on functions of bodies, metabolism and immunity are presented.

An example of assessing the response of a higher education applicant (HEA) to the practical situation of pharmaceutical chemistry

The substance of isoniazid was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of pyridine ring and of hydrazine residue.

Quantitative determination should be carried out in accordance with the method of SPhU 1.2: dissolve 0,250 g in water and dilute to 100,0 ml with the same solvent. To 20,0 ml of the solution add 100 ml of water, 20 ml of hydrochloric acid, 0,2 g of potassium bromide and 0,05 ml of methyl red solution. Titrate dropwise with 0,0167 M potassium bromate, shaking continuously, until the red colour disappears.

1 ml of 0,0167 M potassium bromate is equivalent to 3,429 mg of $C_6H_7N_3O$.

Weight loss on drying is 0,5%.

Isoniazid contains not less than 99,0 per cent and not more than the equivalent of 101,0 per cent of pyridine-4-carbohydrazide, calculated with reference to the dried substance.

Tasks:

1. Identify the substance of isoniazid by functional groups (pyridine ring, hydrazine residue) and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of isoniazid in the substance and draw a conclusion about its quality.

Example of answer and accrual of points:

Parameters that evaluate	**Student response**	**Point**
Reaction of dentification №1. The student offers any of the possible reactions of identification.		**0 / 0,25 / 0,5**
	A) 2,4-dinitrochlorobenzene, sodium hydroxide solution	0,5
	A) a brown-red color appears	0,25
	B) bromine thiocyanate (rodanbromide reagent), sodium hydroxide solution, primary aromatic amine	0,5
	B) a red, yellow or orange.	0,25
Reaction of dentification №2.		**0 / 0,25 / 0,5**
	ammoniac solution of silver nitrate	0,25

	a yellowish precipitate is formed, then metallic silver appears on the walls of the test tube	0,5
Assay		**0 / 0,25/ 0,5/0,75/1**
	bromatometry, direct titration	0,75
	0,0167 M solution of potassium bromate	0,25
	methyl red	0,25
	titrate until the red color disappears	0,5
	$X, \% = \frac{V \cdot K \cdot T \cdot V_{v.f.} \cdot 100 \cdot 100}{m_н \cdot V_а \cdot (100 - \%_{loss})} = \frac{11,3 \cdot 1,000 \cdot 0,003429 \cdot 100 \cdot 100 \cdot 100}{0,250 \cdot 20 \cdot (100 - 0,5)} = 97,35\ \%$	1
	The substance doesn't satisfy the requirememts of SPhU 1.2	0,75
Minimum / maximum point		**0 / 5,0**

List of situations: quality control of medicines of different pharmacological groups (medicines that affect on central nervous, cardiovascular, excretory and coagulation systems, antimicrobial medicines (chemotherapeutic agents, antiseptics and disinfectants); medicines that affect on organ functions, metabolism and immunity).

LIST OF PRACTICAL SITUATIONS

Practical situation №1

The substance of isoniazid was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of pyridine ring and of hydrazine residue.

Quantitative determination should be carried out in accordance with the method of SPhU 1.2: dissolve 0,250 g in water and dilute to 100,0 ml with the same solvent. To 20,0 ml of the solution add 100 ml of water, 20 ml of hydrochloric acid, 0,2 g of potassium bromide and 0.05 ml of methyl red solution. Titrate dropwise with 0,0167 M potassium bromate, shaking continuously, until the red colour disappears (V=14,1 ml).

1 ml of 0,0167 M potassium bromate is equivalent to 3,429 mg of $C_6H_7N_3O$.

Weight loss on drying is 0,5%.

Isoniazid contains not less than 99,0 per cent and not more than the equivalent of 101,0 per cent of pyridine-4-carbohydrazide, calculated with reference to the dried substance.

Tasks:

1. Identify the substance of isoniazid by functional groups (pyridine ring, hydrazine residue) and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of isoniazid in the substance and draw a conclusion about its quality.

Practical situation №2

The substance of benzocaine was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of primary aromatic amino group and ethanol residue.

Quantitative determination should be carried out in accordance with the method of SPhU 1.2: dissolve 0,400 g in a mixture of 25 ml of hydrochloric acid and 50 ml of water, add 3 g of potassium bromide and 0,1 ml of neutral red. Cool in ice water ant titrate slowly with constant stirring, adding 0,1 M sodium nitrite solution (V = 24,1 ml).

1 ml of 0,1 M sodium nitrite is equivalent to 16,52 mg of $C_9H_{11}NO_2$

Weight loss on drying is 0,35%.

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.

Tasks:

1. Identify the substance of benzocaine by functional groups (primary aromatic amino group, ethanol residue) and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of benzocaine in the substance and draw a conclusion about its quality.

Practical situation №3

The substance of potassium iodide was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of potassium ions and iodides.

Quantitative determination should be carried out in accordance with the method of SPhU 1.1: dissolve 1,500 g in water and dilute to 100,0 ml with the same solvent. To 20,0 ml of the solution add 40 ml of hydrochloric acid and titrate with 0,05 M potassium iodate until the colour changes from red to yellow. Add 5 ml of chloroform and continue the titration, shaking vigorously, until the chloroform layer is decolourised (V=17,85 ml).

1 ml of 0,05 M potassium iodate is equivalent to 16,60 mg of KI.

Weight loss on drying is 0,45%.

Potassium iodide contains not less than 99,0 per cent and not more than the equivalent of 100,5 per cent of KI, calculated with reference to the dried substance.

Tasks:

1. Identify the substance of potassium iodide by cations and anions (K^+, I^-) and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of potassium iodide in the substance and draw a conclusion about its quality.

Practical situation №4

The substance of caffeine-sodium benzoate was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of xanthines and benzoate ions.

Quantitative determination should be carried out in accordance with the method of MQC: to 0.1 g add 2-3 ml of ether, 0.2 ml of methyl orange solution and titrate with 0.1 M hydrochloric acid solution with shaking until the color of the aqueous layer is changed(V = 2.55 ml).

1 ml of 0,1 M hydrochloric acid is equivalent to 23,20 mg of caffeine-sodium benzoate.

Weight loss on drying is 0,40%.

The content of sodium benzoate must be not less than 58,0 per cent and not more than 62,0 per cent, calculated with reference to the dried substance.

Tasks:

1. Identify the substance of caffeine-sodium benzoate by xanthines and benzoate ions and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of caffeine-sodium benzoate in the substance and draw a conclusion about its quality.

Practical situation №5

The substance of magnesium sulphate heptahydrate was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of sulphates and magnesium.

Quantitative determination should be carried out in accordance with the method of SPhU 1.1: dissolve 0,450 g in 100 ml of water in a volumetric flask of 500 ml. Dilute to 300,0 ml with water, add 10 ml of ammonium chloride buffer solution pH 10,0 and about 50 mg of mordant black. The solution is heated to 40°C and titrate at this temperature with 0,1 M sodium edetate solution (V = 36,6 ml).

1 ml of 0,1 M sodium edetate is equivalent to 12,04 mg of $MgSO_4$

Weight loss on drying is 0,40%.

Magnesium sulphate heptahydrate contains not less than 99,0 per cent and not more than the equivalent of 100,5 per cent of $MgSO_4$, calculated with reference to the dried substance.

Tasks:

1. Identify the substance of magnesium sulphate heptahydrate by cations and anions (Mg^{2+}, SO_4^{2-}) and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of magnesium sulphate heptahydrate in the substance and draw a conclusion about its quality.

Practical situation №6

The substance of metronidazole was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of nitro group and heterocyclic nitrogen.

Quantitative determination should be carried out in accordance with the method of SPhU 1.2: dissolve 0,150 g in 50 ml of anhydrous acetic acid. Titrate with 0,1 M perchloric acid, determining the end-point potentiometrically (V=8,5 мл).

1 ml of 0,1 M perchloric acid is equivalent to 17,12 mg of $C_6H_9N_3O_3$.

Weight loss on drying is 0,38%.

The content of 2-(2-Methyl-5-nitro-1H-imidazol-1-yl)ethanol must be not less than 99,0 per cent and not more than 101,0 per cent, calculated with reference to the dried substance.

Tasks:

1. Identify the substance of metronidazole by functional groups (nitro group, heterocyclic nitrogen) and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of metronidazole in the substance and draw a conclusion about its quality.

Practical situation №7

The substance of salicylic acid was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of salicylate ions and carboxyl group.

Quantitative determination should be carried out in accordance with the method of SPhU 1.2: dissolve 0,120 g in 30 ml of ethanol (96 per cent) and add 20 ml of water. Titrate with 0,1 M sodium hydroxide, using 0,1 ml of phenol red solution as indicator(V=0,86 мл).

1 ml of 0,1 M sodium hydroxide is equivalent to 13,81 mg of $C_7H_6O_3$

Weight loss on drying is 0,44 %.

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.

Tasks:

1. Identify the substance of salicylic acid by functional groups (salicylate ions, carboxyl group) and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of salicylic acid in the substance and draw a conclusion about its quality.

Practical situation №8

The substance of sodium hydrogen carbonate was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of bicarbonates and sodium.

Quantitative determination should be carried out in accordance with the method of SPhU 1.1: dissolve 1,500 g in 50 ml of carbon dioxide-free water. Titrate with 1 M hydrochloric acid, using 0,2 ml of methyl orange solution as indicator(V=17,50 мл).

1 ml of 1 M hydrochloric acid is equivalent to 84,0 mg of $NaHCO_3$.

Sodium hydrogen carbonate contains not less than 99,0 per cent and not more than the equivalent of 100,0 per cent of $NaHCO_3$.

Tasks:

1. Identify the substance of sodium hydrogen carbonate by cations and anions (bicarbonates and sodium) and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of sodium hydrogen carbonate in the substance and draw a conclusion about its quality.

Practical situation №9

The substance of calcium chloride dihydrate was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of chloride ions and calcium.

Quantitative determination should be carried out in accordance with the method of SPhU 1.1: dissolve 0,280 g in 100 ml of water in a volumetric flask of 500 ml. Dilute to 300,0 ml with water, add 10 ml of sodium hydroxide concentrated solution, 200 mg of calconcarboxylic acid and titrate with 0.1 M sodium edetate (V = 18,5 ml).

1 ml of 0,1 M sodium edetate is equivalent to 14,70 mg of $CaCI_2 \cdot 2H_2O$.

Tasks:

1. Identify the substance of calcium chloride dihydrate by cations and anions (chloride ions and calcium) and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of calcium chloride dihydrate in the substance and draw a conclusion about its quality.

Practical situation №10

The substance of glutamic acid was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of carboxyl group and aliphatic amino group.

Quantitative determination should be carried out in accordance with the method of SPhU 1.0: dissolve 0,130 g in 50 ml of carbon dioxide-free water with gentle heating. Cool. Using 0,1 ml of bromothymol blue solution as indicator, titrate with 0,1 M sodium hydroxide until the colour changes from yellow to blue (V=8,80 мл).

1 ml of 0,1 M sodium hydroxide is equivalent to 14,71 mg of $C_5H_9NO_4$.

Weight loss on drying is 0,44 %.

Glutamic acid contains not less than 98,5 per cent and not more than the equivalent of 100,5 per cent of (2S)-2-aminopentanedioic acid, calculated with reference to the dried substance.

Tasks:

1. Identify the substance of glutamic acid by functional groups (carboxyl group and aliphatic amino group) and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of glutamic acid in the substance and draw a conclusion about its quality.

Practical situation №11

The substance of acetylsalicylic acid was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of salicylate ion and with concentrated sulphuric acid and formaldehyde.

Quantitative determination should be carried out in accordance with the method of MQC: dissolve 0,5 g in 10 ml of alcohol neutralized by phenolphthalein (5-6 drops) and cooled to 8-10°C. Titrate with 0,1 M sodium hydroxide solution (V = 27,2 ml).

1 ml of 0,1 M sodium hydroxide is equivalent to 0,01802 g of acetylsalicylic acid.

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.

Tasks:

1. Identify the substance of acetylsalicylic acid by the presence of salicylate ion and with concentrated acid sulfuric and formaldehyde and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of acetylsalicylic acid in the substance and draw a conclusion about its quality.

Practical situation №12

The substance of ascorbic acid was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions with silver nitrate solution and ferrous sulfate solution.

Quantitative determination should be carried out in accordance with the method of SPhU 2.0: dissolve 0,150 g in a mixture of 10 ml of dilute sulphuric acid and 80 ml of carbon dioxide-free water. Add 1 ml of starch solution. Titrate with 0,05 M iodine until a persistent violet-blue colour is obtained(V =17,0 ml).

1 ml of 0,05 M iodine is equivalent to 8,81 mg of $C_6H_8O_6$.

Ascorbic acid contains not less than 99,0 per cent and not more than the equivalent of 100,5 per cent of (5R)-5-[(1S)-1,2-dihydroxyethyl]-3,4-dihydroxyfuran-2(5H)-one.

Tasks:

1. Identify the substance of ascorbic acid by the reactions with silver nitrate solution and ferrous sulfate solution and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of ascorbic acid in the substance and draw a conclusion about its quality.

Practical situation №13

The substance of metamizole sodium (analginum) was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions with strong hydrogen peroxide solution and on sodium cation.

Quantitative determination should be carried out in accordance with the method of SPhU 2.0: dissolve 0,200 g in 10 ml of 0,01 M hydrochloric acid previously cooled in iced water and titrate immediately, dropwise, with 0,05 M iodine. Before each addition of 0,05 M iodine dissolve the precipitate by swirling. At the end of the titration add 2 ml of starch solution and titrate until the blue colour of the solution persists for at least 2 min. The temperature of the solution during the titration must not exceed 10 °C (V=11,9 ml).

1 ml of 0,05 M iodine is equivalent to 16,67 mg of $C_{13}H_{16}N_3NaO_4S$.

Content: 99,0 per cent to 101,0 per cent (dried substance).

Tasks:

1. Identify the substance of metamizole sodium by the reactions with strong hydrogen peroxide solution and on sodium cation and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of metamizole sodium in the substance and draw a conclusion about its quality.

Practical situation №14

The substance of paracetamol was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions with solution of iron (III) chloride and diazonium salt.

Quantitative determination should be carried out in accordance with the method of MQC: place 0,250 g of in a volumetric flask of 100 ml, add 10 ml of dilute hydrochloric acid and boil with a reflux refrigerator for 1 hour. Then the refrigerator is washed with 30 ml of water, the contents of the flask are quantitatively transferred to a glass for diazotization, wash the flask with 30 ml of water, add 1 g of potassium bromide and titrate with 0,1 M sodium nitrite solution, adding it first at a rate of 2 ml per minute and at the end of titration (before 0,5 ml to the equivalent amount) of 0,05 ml every minute (V = 16,2 ml). The end of the titration is determined with iodine starch paper (exposure 3 minutes).

1 ml of 0,1 M sodium nitrite solution is equivalent to 0,01512 g of paracetamol.

Content: not less than 98,5%.

Tasks:

1. Identify the substance of paracetamol by the reactions with solution of iron (III) chloride and diazonium salt and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of paracetamol in the substance and draw a conclusion about its quality.

Practical situation №15

The substance of sulfacetamide sodium was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of primary aromatic amino group and sodium cation.

Quantitative determination should be carried out in accordance with the method of SPhU 2.0: dissolve 0,500 g in a mixture of 50 ml of water and 20 ml of dilute hydrochloric acid. Cool the solution in iced water and titrate with 0,1 M sodium nitrite solution, adding it first at a rate of 2 ml per minute, and at the end of titration (before 0,5 ml to the equivalent amount) of 0,05 ml every minute (21,1 ml). The end of the titration is determined with iodine starch paper (exposure 3 minutes).

1 ml of 0,1 M sodium nitrite is equivalent to 23,62 mg of $C_8H_9N_2NaO_3S$.

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.

Tasks:

1. Identify the substance of sulfacetamide sodium by the reactions of primary aromatic amino group and sodium cation and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of sulfacetamide sodium in the substance and draw a conclusion about its quality.

Practical situation №16

The substance of phenol was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of phenolic hydroxyl and bromination.

Quantitative determination should be carried out in accordance with the method of SPhU 2.0: dissolve 2,000 g of phenol in *water* 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 bromide-bromate* and 5 ml of *hydrochloric acid*, close the flask, allow to stand with occasional swirling for 30 min. Then allow to stand for 15 min. Add 5 ml of a 200 g/l solution of *potassium iodide*, shake and titrate with *0,1 M sodium thiosulfate* until a faint yellow colour remains. Add 0.5 ml of *starch solution* and 10 ml of *chloroform* and continue the titration with vigorous shaking (V= 3,94 ml). Carry out a blank titration (V_{blank}=35,92 ml).

1 ml of *0.0167 M bromide-bromate* is equivalent to 1.569 mg of C_6H_6O.

Phenol contains not less than 99,0% and not more than the equivalent of 100,5% C_6H_6O.

Tasks:

1. Identify the substance of phenol by the reactions of phenolic hydroxyl and bromination and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of phenol in the substance and draw a conclusion about its quality.

Practical situation №17

The substance of nicotinic acid was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of pyridine ring and carboxylic group.

Quantitative determination should be carried out in accordance with the method of SPhU 2.0: dissolve 0,250 g in 50 ml of water. Add 0,25 ml of phenolphthalein solution. Titrate with 0,1 M sodium hydroxide until a pink colour is obtained (V=20,13ml).

Carry out a blank titration (V=0,2 ml).

1 ml of 0,1 M sodium hydroxide is equivalent to 12,31 mg of $C_6H_5NO_2$.

Content: 99,5 per cent to 100,5 per cent of pyridine-3-carbonic acid (for dried substance, loss on drying is 0,45%).

Tasks:

1. Identify the substance of nicotinic acid by the reactions of pyridine ring and carboxylic group and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of nicotinic acid in the substance and draw a conclusion about its quality.

Practical situation №18

The substance of potassium chloride was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of potassium ions and of chlorides.

Quantitative determination should be carried out in accordance with the method of SPhU 2.0: Dissolve 1,300 g in water and dilute to 100,0 ml with the same solvent. To 10,0 ml of the solution add 50 ml of water, 5 ml of dilute nitric acid, 25,0 ml of 0,1 M silver nitrate and 2 ml of dibutyl phthalate. Shake. Titrate with 0,1 M ammonium thiocyanate, using 2 ml of ferric ammonium sulfate solution as indicator and shaking vigorously towards the end-point (V=13,2 ml).

Loss on drying is 1%.

1 ml of 0,1 M silver nitrate is equivalent to 7,46 mg of KCl.

Content: 99.0 per cent to 100.5 per cent of KCl (dried substance).

Tasks:

1. Identify the substance of potassium chloride by the reactions of potassium ions and of chlorides and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of potassium chloride in the substance and draw a conclusion about its quality.

Practical situation №19

The substance of chloramphenicol (levomycetin) was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of primary amines (after reduction of nitrogroup) and of chlorides.

Quantitative determination should be carried out in accordance with the method of MQC: 0,50 g of substance put into conical flask, add 20 ml of hydrochloric acid concentrated and add 5 g of zinc dust in small portions. After dissolving of zinc dust add again 10 ml of hydrochloric acid concentrated. Trasfer the mixture for diazotation into a beaker, cool with ice-bath. . Add 3 g of potassium bromide and titrate with 0,1 M sodium nitrite (V=15,62 ml).

1 ml of 0,1 M sodium nitrite is equivalent to 0,03231 g of chloramphenicol.

Content: 98,0 per cent to 102,0 per cent of chloramphenicol.

Tasks:

1. Identify the substance of chloramphenicol by the reactions of primary amines (after reduction of nitrogroup) and of chlorides and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of chloramphenicol in the substance and draw a conclusion about its quality.

Practical situation №20

The substance of phenylephrine hydrochloride was received for analysis. It is known that the qualitative analysis yielded positive results in the reactions of phenolic hydroxyl and of chlorides.

Quantitative determination should be carried out in accordance with the method of SPhU 2.0: dissolve 0,150 g in a mixture of 0,5 ml of 0,1 M hydrochloric acid and 80

ml of ethanol (96 per cent). Titrate with 0,1 M ethanolic sodium hydroxide (V=7,25 ml).
1 ml of 0,1 M ethanolic sodium hydroxide is equivalent to 20.37 mg of $C_9H_{14}ClNO_2$.
Content: 98,5 per cent to 101,0 per cent of phenylephrine hydrochloride.
Tasks:
1. Identify the substance of phenylephrine hydrochloride by the reactions of phenolic hydroxyl and of chlorides and indicate their analytical effects.
2. Specify the name of method of quantitative determination, titrant, way of fixing the equivalence point and color transition, calculate the quantitative content of phenylephrine hydrochloride in the substance and draw a conclusion about its quality.