MINISTRY OF PUBLIC HEALTH OF UKRAINE
NATIONAL UNIVERSITY OF PHARMACY

Educational degree *master* (level of educational degree) Training area *22 – PUBLIC HEALTH* (code number and title of training area)

Speciality *226 – PHARMACY, INDUSTRIAL PHARMACY* (code number and title of speciality) Educational program *PHARMACY* (Фм(5,0)англ) (title of educational program)

Semester *spring semester*, 2019/2020 *academic year* Subject *ANALYTICAL CHEMISTRY* (title of academic subject)

EXAM (EXAMPLE)

QUESTION CARD № _____

1. Carry out qualitative analysis of the certain salt. Write the equations of the reactions of cation and anion detection and reactions of cation and anion with the respective group reagents; specify the conditions of their carrying out.

2. Carry out determination of $Na_2C_2O_4$ ($M(Na_2C_2O_4)$ = 134.000 g/mole) by the method of permanganatometry (the method of separate samples). Write the equation of reaction. Calculate the stoichiometrical ratio *s*, the factor of equivalence *f* for the substance to be determined and its molar mass of equivalent *E*. Calculate the percentage of the substance to be determined in three ways – according to the molar mass of equivalent, according to the molar mass and according to the titre of the titrant by the substances to be determined ($c(1/5KMnO_4)$ = 0.1015 mole/dm^3, $V(KMnO_4)$ = 25.18 cm^3, $m(Na_2C_2O_4)$ = 0.2458 g).

3.1. Detection of glucose and maltose has been carried out by the method of thin-layer chromatography. The next distances have been passed by glucose and maltose – 3.8 cm and 2.3 cm respectively. The solvent has passed the distance of 10.0 cm for the same time. Calculate the values of R_f for each substance to be determined.

3.2. Determination of the substance by the method of spectrophotometry. At λ = 410 nm the molar absorption coefficient is 8000 L/mole·cm, the specific absorption coefficient is 147.75 1/%·cm. Calculate the molar mass of the substance to be determined.

3.3. Determination of glucose in the solution by the method of polarimetry. The rotation angle of polarization plane for the solution to be analysed is +11.80º, the layer thickness is 1 dm and the value of the specific rotation is +53.1º. Calculate the concentration of glucose (g/100 mL) in the solution to be analysed.

3.4. Determination of the individual substance by the method of refractometry. Calculate the concentration of the KBr solution, if the refraction index of the solution *n* is equal to 1.3574, the refraction index of the solvent n_0 is equal to 1.3330, the refraction index factor *F* is equal to 0.00117.

POINTS DISTRIBUTION

question 1	**40 points**
question 2	**40 points**
question 3	**20 points**
in all	**100 points**

Estimation scale: national and ECTS

Points in all	ECTS mark	Mark by national scale
90 – 100	**A**	**5**
82 – 89	**B**	**4**
74 – 81	**C**	
64 – 73	**D**	**3**
60 – 63	**E**	
0 – 59	**F**	**2**

It has been approved at the meeting of the Analytical Chemistry Department.
The minutes №1 from 29. 08. 2019 year.

Head of the Analytical Chemistry Department, prof. _____ (sign) I. S. Grytsenko

Examiners, as. prof. _____ (sign) O. Ye. Mykytenko

_____ (sign) L. Yu. Klimenko

performed by as. prof. Klimenko L. Yu., as. prof. Mykytenko O. Ye., as. prof. Kostina T. A.

1. Carry out qualitative analysis of $CoCl_2$. Write the equations of the reactions of cation and anion detection and reactions of cation and anion with the respective group reagents; specify the conditions of their carrying out.

$$CoCl_2 \rightarrow Co^{2+} + 2\ Cl^-$$

1. Action of the group reagent for Co^{2+}-cations – ammonia solution:

$$Co^{2+} + NH_3 \cdot H_2O + Cl^- \rightarrow \underset{\text{dark-blue}}{Co(OH)Cl\downarrow} + NH_4^+$$

$$Co(OH)Cl\downarrow + 6\ NH_3 \cdot H_2O \rightarrow \underset{\substack{\text{orange}\\ \text{solution}}}{[Co(NH_3)_6]^{2+}} + Cl^- + OH^- + 6\ H_2O$$

2. Identification of Co^{2+}-cations:

2.1. Action of ammonium thiocyanate solution:

$$2\ NH_4^+ + Co^{2+} + 4\ SCN^- \rightarrow \underset{\text{dark blue solution}}{(NH_4)_2[Co(SCN)_4]}$$

3. Soda extraction:

$$2\ Co^{2+} + 2\ CO_3^{2-} + H_2O \rightarrow (CoOH)_2CO_3\downarrow + CO_2\uparrow$$

4. Action of the group reagent for Cl^--anions – silver nitrate solution:

$$Cl^- + Ag^+ \rightarrow \underset{\text{white}}{AgCl\downarrow}$$

5. Identification of Cl^--anions:

5.1. Formation of AgCl caseous precipitate and its dissolution in $NH_3 \cdot H_2O$:

$$Cl^- + Ag^+ \rightarrow \underset{\text{white}}{AgCl\downarrow}$$

$$AgCl\downarrow + 2\ NH_3 \cdot H_2O \rightarrow [Ag(NH_3)_2]^+ + Cl^- + 2\ H_2O$$

5.2. Action of potassium dichromate:

The reaction is carried out in a dry way. Chloride-ions interact with $K_2Cr_2O_7$ in the presence of H_2SO_4 and form the volatile substance CrO_2Cl_2:

$$4\ CaCl_2 + K_2Cr_2O_7 + 3\ H_2SO_4 \rightarrow 2\ CrO_2Cl_2\uparrow + 2\ CaSO_4 + K_2SO_4 + 3\ H_2O$$

Gaseous CrO_2Cl_2 is identified by appearance of a red-violet colour of filter paper impregnated with diphenylcarbazide solution.

2. Carry out determination of $Na_2C_2O_4$ ($M(Na_2C_2O_4)$ = 134.000 g/mole) by the method of permanganatometry (the method of separate samples). Write the equation of reaction. Calculate the stoichiometrical ratio *s*, the factor of equivalence *f* for the substance to be determined and its molar mass of equivalent *E*. Calculate the percentage of the substance to be determined in three ways – according to the molar mass of equivalent, according to the molar mass and according to the titre of the titrant by the substances to be determined ($c(1/5KMnO_4)$ = 0.1015 mole/dm³, $V(KMnO_4)$ = 25.18 cm³, $m(Na_2C_2O_4)$ = 0.2458 g).

$$\begin{array}{l|l} -2\,e + C_2O_4^{2-} \rightarrow 2\,CO_2 & 5 \\ +5\,e + MnO_4^- + 8\,H^+ \rightarrow Mn^{2+} + 4\,H_2O & 2 \end{array}$$

$$5\,C_2O_4^{2-} + 2\,MnO_4^- + 16\,H^+ \rightarrow 10\,CO_2 + 2\,Mn^{2+} + 8\,H_2O$$

$s = 5/2$; $f(Na_2C_2O_4) = 1/2$; $f(KMnO_4) = 1/5$;

$E(Na_2C_2O_4) = M(Na_2C_2O_4)\cdot f(Na_2C_2O_4) = 134.000\cdot 1/2 = 67.000$ g/mole

Given:

$c(1/5KMnO_4)$ = 0.1015 mole/dm³
$V(KMnO_4)$ = 25.18 cm³
$m(Na_2C_2O_4)$ = 0.2458 g
$E(Na_2C_2O_4)$ = 67.000 g/mole
$M(Na_2C_2O_4)$ = 134.000 g/mole

$\omega(Na_2C_2O_4)$ – ?

calculation of $\omega(Na_2C_2O_4)$ according to $E(Na_2C_2O_4)$

$$\omega(Na_2C_2O_4) = \frac{c(1/5KMnO_4)\cdot V(KMnO_4)\cdot E(Na_2C_2O_4)\cdot 100}{1000\cdot m(Na_2C_2O_4)} =$$

$$= \frac{0.1015\cdot 25.18\cdot 67.000\cdot 100}{1000\cdot 0.2458} = 69.70\%$$

$$c(KMnO_4) = c(1/5KMnO_4)\cdot f(KMnO_4) = 0.1015\cdot 1/5 = 0.02030 \text{ mole/dm}^3$$

calculation of $\omega(Na_2C_2O_4)$ according to $M(Na_2C_2O_4)$ and *s*

$$\omega(Na_2C_2O_4) = \frac{c(KMnO_4)\cdot V(KMnO_4)\cdot s\cdot M(Na_2C_2O_4)\cdot 100}{1000\cdot m(Na_2C_2O_4)} =$$

$$= \frac{0.02030\cdot 25.18\cdot 5/2\cdot 134.000\cdot 100}{1000\cdot 0.2458} = 69.70\%$$

calculation of $\omega(Na_2C_2O_4)$ according to $T(KMnO_4/Na_2C_2O_4)$

$$T(KMnO_4/Na_2C_2O_4) = \frac{c(1/5KMnO_4)_{theor}\cdot E(Na_2C_2O_4)}{1000} = \frac{0.1000\cdot 67.000}{1000} = 0.006700 \text{ g/cm}^3$$

$$T(KMnO_4/Na_2C_2O_4) = \frac{c(KMnO_4)_{theor}\cdot s\cdot M(Na_2C_2O_4)}{1000} = \frac{0.02000\cdot 5/2\cdot 134.000}{1000} = 0.006700 \text{ g/cm}^3$$

$$K(KMnO_4) = \frac{c(1/5KMnO_4)_{pract}}{c(1/5KMnO_4)_{theor}} = \frac{0.1015}{0.1000} = 1.015$$

$$\omega(Na_2C_2O_4) = \frac{K(KMnO_4)\cdot V(KMnO_4)\cdot T(KMnO_4/Na_2C_2O_4)\cdot 100}{m(Na_2C_2O_4)} =$$

$$= \frac{1.015\cdot 25.18\cdot 0.006700\cdot 100}{0.2458} = 69.70\%$$

performed by as. prof. Klimenko L. Yu., as. prof. Mykytenko O. Ye., as. prof. Kostina T. A.

3.1. Detection of glucose and maltose has been carried out by the method of thin-layer chromatography. The next distances have been passed by glucose and maltose – 3.8 cm and 2.3 cm respectively. The solvent has passed the distance of 10.0 cm for the same time. Calculate the values of R_f for each substance to be determined.

Given:

$S(glucose) = 3.8$ cm $S(maltose) = 2.3$ cm $S(solvent) = 10.0$ cm	$R_f(glucose) = \frac{S(glucose)}{S(solvent)} = \frac{3.8}{10.0} = 0.38$
$R_f(glucose)$ – ? $R_f(maltose)$ – ?	$R_f(maltose) = \frac{S(maltose)}{S(solvent)} = \frac{2.3}{10.0} = 0.23$

3.2. Determination of the substance by the method of spectrophotometry. At λ = 410 nm the molar absorption coefficient is 8000 L/mole·cm, the specific absorption coefficient is 147.75 1/%·cm. Calculate the molar mass of the substance to be determined.

Given:

$\varepsilon = 8000$ L/mole·cm $A_{1cm}^{1\%} = 147.751/\%\cdot cm$	$M = \frac{\varepsilon}{A_{1cm}^{1\%}} \cdot 10 = \frac{8000}{147.75} \cdot 10 = 541.46$ g/mole
M – ?	

3.3. Determination of glucose in the solution by the method of polarimetry. The rotation angle of polarization plane for the solution to be analysed is +11.80º, the layer thickness is 1 dm and the value of the specific rotation is +53.1º. Calculate the concentration of glucose (g/100 mL) in the solution to be analysed.

Given:

$\alpha = +11.80°$ $[\alpha]_D^{20} = +53.1°$	$C = \frac{\alpha \cdot 100}{[\alpha]_D^{20} \cdot l} = \frac{11.80 \cdot 100}{53.1 \cdot 1} = 22.2$ g/100 mL
C – ?	

3.4. Determination of the individual substance by the method of refractometry. Calculate the concentration of the KBr solution, if the refraction index of the solution n is equal to 1.3574, the refraction index of the solvent n_0 is equal to 1.3330, the refraction index factor F is equal to 0.00117.

Given:

$n = 1.3574$ $n_0 = 1.3330$ $F(KBr) = 0.00117$	$C(KBr) = \frac{n - n_0}{F(KBr)} = \frac{1.3574 - 1.3330}{0.00117} = 20.85\%$
$C(KBr)$ – ?	