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)

FINAL MODULE CONTROL
MODULE 2. CLASSIC QUALITATIVE ANALYSIS. INSTRUMENTAL METHODS OF ANALYSIS

QUESTION CARD (EXAMPLE)

1. **Propose at least three methods of quantitative determination for FeI_2 by titrimetric methods of analysis. For each method specify the titrant and indicator. Write the equation of reaction. Calculate the stoichiometrical ratio *s* and factor of equivalence *f* for the substance to be determined.**
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.**

4. **Answer the tests.**

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FINAL MODULE CONTROL
MODULE 2. CLASSIC QUALITATIVE ANALYSIS. INSTRUMENTAL METHODS OF ANALYSIS

QUESTION CARD (EXAMPLE)

POINTS DISTRIBUTION

question 1	**12 points**
question 2	**12 points**
question 3	**6 points**
question 4	**10 points**
in all	**40 points**

Estimation scale: national and ECTS

Points in all	ECTS mark	Mark by national scale
36 – 40	**A**	**5**
34 – 35	**B**	**4**
30 – 33	**C**	
26 – 29	**D**	**3**
24 – 25	**E**	
0 – 23	**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. _____ I. S. Grytsenko
(sign)

Examiner, as. prof. _____ L. Yu. Klimenko
(sign)

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

FINAL CONTROL №2

1. Propose at least three methods of quantitative determination for FeI_2 by titrimetric methods of analysis. For each method specify the titrant and indicator. Write the equation of reaction. Calculate the stoichiometrical ratio *s* and factor of equivalence *f* for the substance to be determined.

1) cerimetry:

$- e + Fe^{2+} \rightarrow Fe^{3+}$	1
$+ e + Ce^{4+} \rightarrow Ce^{3+}$	1
$Fe^{2+} + Ce^{4+} \rightarrow Fe^{3+} + Ce^{3+}$	

$s = 1$; $f(FeI_2) = 1$

titrant – $Ce(SO_4)_2$; indicator – ferroin

2) bromatometry:

$- e + Fe^{2+} \rightarrow Fe^{3+}$	6
$+ 6 e + BrO_3^- + 6 H^+ \rightarrow Br^- + 3 H_2O$	1
$6 Fe^{2+} + BrO_3^- + 6 H^+ \rightarrow 6 Fe^{3+} + Br^- + 3 H_2O$	

$s = 6$; $f(FeI_2) = 1$

titrant – $KBrO_3$; indicator – methyl orange

3) permanganatometry:

$- e + Fe^{2+} \rightarrow Fe^{3+}$	5
$+ 5 e + MnO_4^- + 8 H^+ \rightarrow Mn^{2+} + 4 H_2O$	1
$5 Fe^{2+} + MnO_4^- + 8 H^+ \rightarrow 5 Fe^{3+} + Mn^{2+} + 4 H_2O$	

$s = 5$; $f(FeI_2) = 1$

titrant – $KMnO_4$; without an indicator

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

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).

$-2\ e + C_2O_4^{2-} \rightarrow 2\ CO_2$	5
$+5\ e + MnO_4^- + 8\ H^+ \rightarrow Mn^{2+} + 4\ H_2O$	2

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

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

$E(Na_2C_2O_4) = M(Na_2C_2O_4) \cdot f(Na_2C_2O_4) = 134.000 \cdot 1/2 = 67.000$ 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 $T(KMnO_4/Na_2C_2O_4)$

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

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

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

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

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

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:

ε = 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:

α = +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:

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