Ministry of Health of the Republic of Belarus
Educational institution
"Gomel State Medical University"

Department of General and Bioorganic Chemistry

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METHODOLOGICAL MANUAL
for conducting the laboratory class
in the academic discipline "Medical chemistry"
for students
the first-year of the Faculty of International Students (FIS),
studying in the specialty 7-07-0911-01 "Medical business"

Topic 3: Acid-base titration

Time: 2 hours

Approved at the meeting of the Department
of General and Bioorganic Chemistry
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THE TRAINING AND EDUCATIONAL GOAL, TASKS, MOTIVATION TO STUDY THE TOPIC

Training purpose:

– formation of students' basic professional competence for solving diagnostic, research and other tasks of professional activity based on knowledge about the principles of the acid-base titration method;

– formation of skills and abilities to work with measuring utensils, the ability to perform calculations of the content of the analyzed substance according to the results of titration;

– familiarization with the role of acid-base titration in biomedical research and clinical analysis.

Educational goal:

– to develop your personal, spiritual potential;

– to form the qualities of a patriot and a citizen who is ready to actively participate in the economic, industrial, socio-cultural and social life of the country;

– learn to observe academic and labor discipline, the norms of medical ethics and deontology;

– to realize the social significance of their future professional activities.

Tasks:

As a result of the training session, the student should

know:

– the theoretical bases of acid-base titration (neutralization method); types of acid-base titration (acidimetry and alkalimetry);

– acid-base indicators, indicator selection during analysis;

– the role of acid-base titration methods in biomedical research and clinical analysis;

be able to:

– perform the typical calculating based on the Equivalent Law involving the rules of significant figures;

– choose the type of acid-base titration, working solution, standard solution, method of fixing the equivalence point during analysis;

– apply methods of statistical processing of the experimental data

possess:

– skills in the preparation and standardization of hydrochloric acid solutions of a certain concentration;

– skills of performing of acid-base titration.

Motivation to study the topic:

Acid-base titration methods are widely used in biomedical research, they help to solve many problems that arise during the chemical analysis of biological fluids both during diagnosis and in the treatment of patients.

In clinical laboratories, acid-base titration is used to determine the acidity of gastric juice, the buffer capacity of blood, cerebrospinal fluid, etc. Using this method, drugs are analyzed, the quality of food products (milk, bread, flour, etc.) is established. The neutralization method has a great importance in the sanitary and hygienic assessment of the state of the environment (natural reservoirs, soil, industrial effluents, etc.).

NECESSARY EQUIPMENT

1. Methodological manual for students on the topic "Acid-base titration".
2. Training tables:
a) Periodic Table of chemical elements by D.I. Mendeleev;
b) table of solubility of acids, bases and salts.
3. Reference materials of basic physico-chemical constants.
4. Chemical reagents and equipment necessary for laboratory work.

CONTROL QUESTIONS ON THE TOPIC OF THE CLASS

1. The theoretical bases of acid-base titration.
2. Acid-base indicators.
3. Calculations in the acid-base titration method.

COURSE OF THE CLASS

The theoretical part

1. THE THEORETICAL BASES OF ACID-BASE TITRATION

Acid-base titration is one of the titration methods based on a neutralization reaction:

$$\mathbf{H^+ + OH^- \rightarrow H_2O}$$

Acid-base reactions (neutralization reactions) are characterized by a high rate and run strictly stoichiometrically. The acid-base titration method is used for the quantitative determination of acids (H_2SO_4, HNO_3, HCl, H_3PO_4, CH_3COOH, $H_2C_2O_4$), bases (KOH, NaOH, $Ba(OH)_2$), as well as salts that can be hydrolyzed in aqueous solutions (Na_2CO_3, K_2SO_3, $MgCl_2$, $ZnSO_4$).

Figure 1 shows the scheme of the titration unit for acid-base titration.

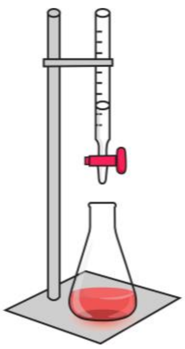

Figure 1 – Setup for a typical acid-base titration

Depending on the nature of the titrant used, acid-base titration is divided into:

1) acidimetry;

• *working solutions:* HCl, H_2SO_4 solutions;
• *primary standards:* solutions of $Na_2B_4O_7 \times 10H_2O$ (borax), $Na_2CO_3 \times 10H_2O$;
• *test solutions:* solutions of strong and weak bases, salts which provide the basic medium as a result of their hydrolysis (Na_2CO_3, K_2S);

2) alkalimetry;

• *working solutions:* NaOH, KOH solutions;

• *primary standards:* solutions of $H_2C_2O_4 \times 2H_2O$ (oxalic acid) and $H_2C_4H_4O_4$ (succinic acid);

• *test solutions:* solutions of strong and weak acids, salts which provide the acidic medium as a result of their hydrolysis ($MgCl_2$, $Zn(NO_3)_2$).

The endpoint or equivalence point for acid-base titration is determined by the color change of the acid-base indicator [1-3].

2. ACID-BASE INDICATORS

Reactions between acids and bases are not accompanied, as a rule, by any external effects, therefore, special indicator substances have to be used to fix the equivalence point.

Acid-base indicators are either weak organic acids or weak organic bases, conjugate forms of which have a different colors. The equilibrium in a solution of the acid-base indicator, for example, a weak acid, can be represented by the following equation in which HInd is as a simple representation for the complex indicator molecule:

$$\underset{\text{color 1}}{HInd} \rightleftarrows H^+ + \underset{\text{color 2}}{Ind^-}$$

Thus, the molecular HInd and ionic Ind^- forms of the indicator have different colors, and the ratio of concentrations of HInd and Ind^- forms depends on the pH value.

If acid is added to the solution with the indicator, the increased H^+ concentration shifts the equilibrium towards the non-ionized form, which is responsible for color 1, in accordance with Le Châtelier's principle. If to add base, the equilibrium will shift towards the ionized form which is responsible for color 1.

The color change of the acid-base indicator depends on the ratio of [HInd] to [Ind^-].
In general: when $[HInd] / [In^-] > 10$, color 1 dominates
when $[HInd] / [In^-] < 10$, color 2 dominates

The color change of the acid-base indicator does not occur at a fixed pH value, but occurs in the pH interval, called the pH range. As a rule, it is noted that the color change takes place in the range:

$$\mathbf{pH = pK_{in} \pm 1}$$

Table 1 – Some common acid-base indicators

Name of indicator	**Color of indicator**		**pH rage***	**pK$_{in}$**
	in acidic medium	**in basic medium**		
Thymol blue	red	yellow	1.2–2.8	1.7
Bromophenol blue	yellow	bluish purple	3.0–4.6	3.8
Methyl orange	orange	yellow	3.1–4.4	4.0
Methyl red	red	yellow	4.2–6.3	5.5
Chlorophenol blue	yellow	red	4.8–6.4	5.6
Bromothymol blue	yellow	blue	6.0–7.6	7.0
Cresol red	yellow	red	7.2–8.8	7.6
Phenolphthalein	colorless	reddish pink	8.3–10.0	9.0

* The pH range is defined as a range within which an indicator changes the acid color to the base color.

The choice of the indicator depends upon the reaction that needs to be performed. To complete the titration correctly, the pH range of the indicator must correspond to the pH value at the endpoint [1, 2].

In analytical practice, when titrating strong acids with a strong base, methylorange and phenolphthalein are most often used; when titrating a weak base with a strong acid, methylorange; when titrating a weak acid with a strong base, phenolphthalein. In the presence of two acids, two bases or two hydrolyzed salts in the solution, two equivalence points are fixed using two indicators.

3. CALCULATIONS IN THE ACID-BASE TITRATION METHOD

Calculations in the acid-base titration method are based on the Equivalent Law. At the equivalence point, the number moles of the equivalent of the analyte is equal to the number moles of the equivalent of titrant:

$$n_e(A) = n_e(T)$$

In practice, the concentration of the analyte or its mass can be calculated using the following formulas:

1) $C_N(A) \times V(A) = C_N(T) \times V(T)$; **2)** $\frac{m(A)}{M_e(A)} = \frac{C_N(T) \times V(T)}{1000}$

Problem 1. 12.5 ml of 0.5000 M H_2SO_4 solution was used to neutralize 50.0 ml of KOH solution. Calculate the normal concentration of KOH in the solution, given that $f_e(H_2SO_4) = ½$ in this reaction.

Solution:

$2KOH + H_2SO_4 \rightarrow K_2SO_4 + 2H_2O$

According to the Equivalent Law: $n_e(KOH) = n_e(H_2SO_4)$

Since $C_N(KOH) \times V(KOH) = C_N(H_2SO_4) \times V(H_2SO_4)$

$$C_N(KOH) = \frac{V(H_2SO_4) \times C_N(H_2SO_4)}{V(KOH)};\ C_N(H_2SO_4) = \frac{C_M(H_2SO_4)}{f_e(H_2SO_4)} = \frac{0.5000}{0.5} = 1.0000N$$

$$C_N(KOH) = \frac{12.5 \times 1.0000}{50.0} = 0.2500\,N$$

Answer: $C_N(KOH) = 0.2500\ N$

The practical part

Safety instructions before laboratory work.

Laboratory work

Determination of potassium hydroxide mass in the test solution

Analytical problem: an unknown volume of the analyzed KOH solution is contained in a 50 ml volumetric flask.

1. This procedure is based on the following chemical reaction:

$$KOH + HCl \rightarrow KCl + H_2O$$

2. Choice of the indicator: titration should be carried out in the presence of phenolphthalein, since the initial solution having a basic medium becomes neutral at the equivalence point, what allows us to observe the changing of a reddish pink color of an initial solution into a colorless one.

3. Carry out an experiment in the following sequence of operations:

a) take the analyzed KOH solution of an unknown volume contained in a 50 ml volumetric flask and add distilled water to the mark to get the exact volume of the analyzed solution (V_{overal} = 50 ml);

b) take an analytical pipette and pour 10 ml of the solution into 3 flasks for titration;

c) add two drops of phenolphthalein into each flask;

d) pour HCl solution into a burette up to the zero mark;

i) titrate KOH solution against hydrochloric acid up to the endpoint *when pink color of an initial solution turns colorless* after adding one drop of HCl;

f) repeat such titration two times stating from zero mark of burette; the results must not differ greatly from each other;

j) fill in the table 1 below with the obtained data.

Table 2 – The results of KOH solution titration against HCl solution

№	V(KOH), ml	V(HCl), ml	C_N(KOH), mol/l	T(KOH), g/ml	m(KOH), g
1	10.0				
2	10.0				
3	10.0				

4. Calculation of the results of the experiment:

According to the Equivalent Law in equivalence point: $n_e(HCl) = n_e(KOH)$;

$$C_N(HCl) \times V(HCl) = C_N(KOH) \times V(KOH)$$

- $C_N(KOH) = \frac{C_N(HCl) \times V(HCl)}{V(KOH)}$; $V(KOH) = 10\ ml$
- $T(KOH) = \frac{C_N(KOH) \times M_e(KOH)}{1000}$; $M_e(KOH) = 56\ g/mol$
- $m(KOH) = T(KOH) \times V_{overal}(KOH)$; $V_{overal}(KOH) = 50\ ml$

Sample 1:	**Sample 2:**	**Sample 3:**
$C_N(KOH)_1 = \frac{C_N(HCl) \times V(HCl)_1}{V(KOH)}$	$C_N(KOH)_2 = \frac{C_N(HCl) \times V(HCl)_2}{V(KOH)}$	$C_N(KOH)_3 = \frac{C_N(HCl) \times V(HCl)_3}{V(KOH)}$
$T(KOH)_1 = \frac{C_N(KOH)_1 \times M_e(KOH)}{1000}$	$T(KOH)_2 = \frac{C_N(KOH)_2 \times M_e(KOH)}{1000}$	$T(KOH)_3 = \frac{C_N(KOH)_3 \times M_e(KOH)}{1000}$
$m(KOH)_1 = T(KOH)_1 \times V_{ov}(KOH)$	$m(KOH)_2 = T(KOH)_2 \times V_{ov}(KOH)$	$m(KOH)_3 = T(KOH)_3 \times V_{ov}(KOH)$

5. Statistic treatment of the experimental data:

5.1. The average value of KOH mass:

$$\overline{m}(KOH) = \frac{m_1 + m_2 + m_3}{3} = x.xxxx$$

5.2. The deviation from the average value:

$$d_1 = m_1 - \overline{m}(KOH) = x.xxxx$$
$$d_2 = m_2 - \overline{m}(KOH) = x.xxxx$$
$$d_3 = m_3 - \overline{m}(KOH) = x.xxxx$$

5.3. Measurement variance (s^2) and measurement standard deviation (s):

$$s^2 = \frac{d_1^2 + d_2^2 + d_3^2}{n \times (n-1)} = \qquad s = \sqrt{s^2} =$$

5.4 Confidence interval: $\varepsilon_\alpha = t \times s = 4.30 \times s = x.xxxx$

t is a Student's-t number taken from a statistical table and depending from a chosen level of confidence and a number of measurements; t = 4.30 for a 95 % confidence level and 3 of measurements.

5.5 The result of the KOH mass determination must be given in the following way:
$m(KOH) = \overline{m}(KOH) \pm \varepsilon_\alpha = x.xxxx \pm x.xxxx$ (g)

Control over the assimilation of the topic

It is conducted in the form of written independent work of students.

METHODOLOGICAL RECOMMENDATIONS FOR THE ORGANIZATION AND PERFORMING OF INDEPENDENT WORK OF STUDENT (IWS)

The time allotted for independent work can be used by students for:
– preparation for laboratory classes;
– taking notes of educational literature;
– performing tasks for self-control of knowledge;
– preparation of thematic reports, abstracts, presentations.

The main methods of organizing independent work:
– studying topics and problems that are not covered in the classroom;
– writing an abstract and making a presentation;
– performing tasks for self-control of knowledge.

List of tasks of IWS:

1. In the acid-base titration method, solutions of the following substances can be used as titrants:

a) NH_4OH; b) H_2SO_4; c) $H3PO_4$; d) NaOH.

2. In the acid-base titration method, the following substances can be used to prepare standard solutions:

a) $H_2C_2O_4 \times 2H_2O$; b) $Na_2CO_3 \times 10H_2O$; c) $Na_2B_4O_7 \times 10H_2O$; d) $Na_2SO_4 \times 10H_2O$.

3. To determine the acidity of dairy products as working solutions, you can use:

a) a solution of hydrochloric or sulfuric acid;
b) a solution of sodium hydroxide or potassium hydroxide;
c) a solution of iron (II) hydroxide;
d) a solution of phosphoric acid.

4. To determine the alkali content in the detergent, the following solutions can be used as working solutions:
a) phosphoric acid; c) sulfuric acid;
b) sodium or potassium hydroxide; d) hydrochloric acid.

5. In aqueous solutions of which salts is the reaction of the medium basic?

a) $CaCl_2$; b) $Al_2(SO_4)_3$; c) $NaNO_2$; d) Na_2CO_3.

6. In solutions of which salts is the reaction of the medium acidic?

a) K_2S; b) $BiCl_3$; c) NH_4NO_3; d) KI.

7. The titer of HCl (g/ml) in 0.1 M hydrochloric acid solution is equal to:

a) 0.0073; b) 0.001825; c) 0.09125; d) 0.00365.

8. What volume of hydrochloric acid solution with the mass fraction of HCl equal to 18 % (density is 1.09 g/ml) should be taken to prepare 1 l of 5 N solution of HCl. What is the titer of the solution?

Answer: 930 ml; 0.1825 g/ml

9. 24.10 ml of HCl solution was used for titration of 0.2860 g of $Na_2CO_3 \times 10H_2O$ in the presence of methyl orange. Calculate the molar concentration and the titer of the HCl solution.

Answer: 0.08299 mol/l; 0.003029 g/ml

10. 23.04 ml of NaOH solution was used for titration of 20.00 ml of HCl solution with a titer equal to 0.001825 g/ml. Calculate the normality and titer of the NaOH solution.

Answer: 0.04 mol/l; 0.001600 g/ml

The control of the IWS is carried out in the form of:
– evaluation of an oral answer to a question, message, report or presentation;
– individual conversation.

LIST OF SOURCES USED

1. Основы химии для иностранных студентов = Essential chemistry for foreign students : учебно-методическое пособие / С. В. Ткачёв [и др.]. – 5-е изд. – Минск : БГМУ, 2018. – 168 с. – Режим доступа: http://rep.bsmu.by:8080/handle/BSMU/21054.

2. Филиппова, В. А. Общая химия : учеб. пособие для студентов лечеб. фак-та, обуч. на англ. яз. : в 2 ч. = General Chemistry : Educational guaidance for students medical department in English medium / В. А. Филиппова, А. В. Лысенкова, Л. В. Чернышева. – Гомель : ГомГМУ, 2009. – Ч. 1. – 192 с. URI: https://elib.gsmu.by/handle/GomSMU/2679.

3. Chang, Raymond. Chemistry / R. Chang. – 4th ed. – USA : University Science Books, 1991. – 1065 p.