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METROLOGICAL STUDY OF THE EFFECT OF TEMPERATURE ON THE DISSOCIATION OF ACETIC ACID

This article is devoted to the study of the dissociation reaction of acetic acid at a temperature change in the range from room (20 °C) to 75 °C. In the course of research, the methods were considered, the classification of the considered methods was carried out, and the methodology of the experiments was formulated. The selected technique reflects the express measurement of the hydrogen *pH* indicator using a portable *pH*-meter. Experiments were carried out in laboratory conditions – Lincoln Park, Chicago, USA. Acetic acid with a concentration of 6 mol/l was chosen as the basis. By adding a distilled water, a base concentration of 1 mol/l was obtained. Nodal temperature points were selected for measurements (four points in the temperature range of 20 °C – 75 °C); five experimental samples of acetic acid (1 mol/l) were formed; the analysis of the measurement results at nodal points was carried out for the accuracy of the measurement results of five test samples of acetic acid using first- and second-order statistical moments (mathematical expectation and variance); accuracy characteristics of experimental data (instrumental and methodical errors) were estimated. Research samples (acetic acid samples) were brought to the nodal points with a positive temperature gradient using a steam bath. The measurement error estimate was determined by the accuracy class of the device and was 0.1%. The obtained *pH* values were converted to the number of hydrogen cations, followed by the determination of the degree of dissociation and the dissociation constant. These determinations were carried out under the condition of ensuring chemical equilibrium. The nature of the behavior of the degree and constant of dissociation when the temperature of the test samples changes is clearly non-linear. In the course of research, the main measurement errors were established, the main of which is the nonlinearity of the transformation. Quantitative values of nonlinearity errors were determined by the method of measurements with multiple observations using the Student's correction factor. The article provides conclusions based on the results of research and presents the prospects for temperature correction of *pH*-meters to eliminate the temperature component of the error of *pH*-meters.

Keywords: acetic acid, concentration, *pH*-meter, dissociation, degree of dissociation, dissociation coefficient, temperature, nonlinearity, error, accuracy class

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МЕТРОЛОГІЧНЕ ВИВЧЕННЯ ВПЛИВУ ТЕМПЕРАТУРИ НА ДИСОЦІАЦІЮ ОЦТОВОЇ КИСЛОТИ

Дана стаття присвячена дослідженню реакції дисоціації оцтової кислоти при зміні температури в діапазоні від кімнатної (20 °C) до 75 °C. У ході досліджень було розглянуто методи, виконано класифікацію розглянутих методів та сформульовано методику реалізації експериментів. Вибрана методика відображає експрес-вимірювання водневого показника *pH* за допомогою портативного *pH*-метра. Досліди здійснювались в лабораторних умовах – Лінкольн Парк, Чикаго, США. За основу була обрана оцтова кислота концентрації 6 моль/л. Шляхом додавання дистильованої води отримано базову концентрацію 1 моль/л. Для проведення вимірювань було відібрано вузлові температурні точки (чотири точки в діапазоні температур 20 °C – 75 °C); було сформовано п'ять дослідних зразків оцтової кислоти (1 моль/л); проведено аналіз результатів вимірювання у вузлових точках на предмет кучності результатів вимірювання п'ятих дослідних зразків оцтової кислоти з використанням статистичних моментів першого та другого порядку (математичне очікування та дисперсія); оцінено точнісні характеристики експериментальних даних (інструментальні та методичні похибки). Виведення на вузлові точки зразків дослідження (зразків оцтової кислоти) здійснювались з додатним градієнтом по температурі за допомогою парової бані. Оцінка похибки вимірювань визначалася класом точності приладу і становила 0.1 %. Отримані значення *pH*, перетворені на кількість катіонів водню, з подальшим визначенням ступеня дисоціації та константи дисоціації. Дані визначення здійснювались із умови забезпечення хімічної рівноваги. Характер поведінки ступеня та константи дисоціації при зміні температури дослідних зразків має явно нелінійний характер. У ході досліджень встановлені основні похибки вимірювань, основним з яких є похибка нелінійності перетворення. Кількісні значення похибок нелінійності визначалися методом вимірювань із багаторазовими спостереженнями з використанням поправного коефіцієнта Стюдента. У статті наведено висновки за результатами досліджень та подано перспективи температурної корекції *pH*-метрів для усунення температурної складової похибки роботи *pH*-метрів.

Ключові слова: оцтова кислота, концентрація, *pH*-метр, дисоціація, ступінь дисоціації, константа дисоціації, температура, нелінійність, похибка, клас точності

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МЕТРОЛОГИЧЕСКОЕ ИЗУЧЕНИЕ ВЛИЯНИЯ ТЕМПЕРАТУРЫ НА ДИССОЦИАЦИЮ УКСУСНОЙ КИСЛОТЫ

Данная статья посвящена исследованию реакции диссоциации уксусной кислоты при изменении температуры в диапазоне от комнатной (20°C) до 75°C. В ходе исследований были рассмотрены методы, выполнена классификация рассмотренных методов и сформулирована методика реализации экспериментов. Выбранная методика отражает экспресс-измерение водородного показателя *pH* с помощью портативного *pH*-метра. опыты проводились в лабораторных условиях – Линкольн Парк, Чикаго, США. За основание была выбрана уксусная кислота концентрации 6 моль/л. Путем добавления дистиллированной воды получена базовая концентрация 1 моль/л. Для проведения измерений были отобраны узловые температурные точки (четыре точки в диапазоне температур 20 – 75 °C); было сформировано пять опытных образцов уксусной кислоты (1 моль/л); проведен анализ результатов измерения в узловых точках на предмет кучности результатов измерения пяти опытных образцов уксусной кислоты с использованием статистических моментов первого и второго порядка (математическое ожидание и дисперсия); оценены точные характеристики экспериментальных данных (инструментальные и методические погрешности). Выводы на узловые точки образцов исследования (образцов уксусной кислоты) осуществлялись с положительным градиентом по температуре с помощью паровой бани. Оценка погрешности измерений определялась классом точности и составляла 0.1 %. Получены значения *pH*, превращенные в количество катионов водорода, с последующим определением степени диссоциации и константы диссоциации. Данные определения осуществлялись из условия обеспечения химического равновесия. Характер поведения степени и константы диссоциации при изменении температуры опытных образцов носит явно нелинейный характер. В ходе исследований установлены основные погрешности измерений, основным из которых является погрешность нелинейности превращения. Количественные значения погрешностей нелинейности определялись методом измерений с многократными наблюдениями с использованием поправочного коэффициента Стюдента. В статье приведены выводы по результатам исследований и представлены перспективы температурной коррекции *pH*-метров для устранения температурной составляющей погрешности *pH*-метров.

Ключевые слова: уксусная кислота, концентрация, *pH*-метр, диссоциация, степень диссоциации, константа диссоциации, температура, нелинейность, погрешность, класс точности

The aim and objectives of the study. Acetic acid is one of the most common in production and at home [1]. The use of this acid is very diverse. In the chemical industry, it is used to produce plastics, dyes, medicines, artificial fiber, etc. In the food industry, acetic acid is a preservative, acidity regulator and food additive. Salts of acetic acid are used for primary etching when dyeing fabrics. Acetic acid is widely used as a solvent.

The wide distribution of acetic acid in production (for various industries) and in everyday life determines the external conditions for the presence of this acid. External conditions of stay are, first of all, characterized by environmental parameters.

One of the most common environmental parameters is temperature [2]. Assessing the effect of temperature on the concentration of acetic acid CH_3COOH in mol/l is quite relevant. Moreover, studies on this issue have not been identified in the reference literature. It was only possible to determine that CH_3COOH , acetic acid bacteria, acetic acid and foreign microorganisms die when acetic acid is heated to critical temperatures (110°C).

The purpose of these studies is to determine the effect of temperature changes on the concentration of acetic acid CH_3COOH in mol/l .

To achieve this goal, it is necessary to set and solve a number of tasks. In particular:

- formulate the initial parameters (concentration) of acetic acid;
- determine the temperature range for research;
- set the number of temperature points to measure the concentration of acetic acid in the selected temperature range;
- formulate requirements for the laboratory where research will be carried out;
- select basic methods for determining the concentration of acetic acid;
- determine the basic and auxiliary equipment when carrying out measurements;
- develop methods for determining the concentration of acetic acid; methods should be based on the selected basic methods;
- conduct experiments to determine the concentration of acetic acid;
- process the obtained experimental data;
- formulate conclusions and prospects for further research.

These and other problems need to be solved during research.

Methods and equipment. Successful solution of set goals and objectives requires analysis of possible methods, selection of equipment and development of experimental techniques.

The variety of methods of analysis and synthesis that are used in chemical research can be reduced to some basic ones. These basic methods need to be considered for the further construction of methods that will help solve the problems.

The implementation of methods, as a rule, occurs in laboratory conditions using specialized equipment [3].

The main positions regarding equipment also require detailed consideration.

The main methods used in experimental studies are the following:

- 1) Titration method, which is based on the expression:

$$C_{\kappa} \cdot V_{\kappa} = C_{\text{ш}} \cdot V_{\text{ш}}, \quad (1)$$

where C_{κ} – acid concentration;

V_{κ} – acid volume;

$C_{\text{ш}}$ – alkali concentration;

$V_{\text{ш}}$ – alkali volume.

Formula (1) determines the equilibrium of the concentration of hydrogen cations $C[H^+]$ when mixing acid and alkali.

- 2) A method for producing acid solutions with different mass fractions is necessary to obtain acid solutions of different concentrations by mixing with a neutral liquid. Distilled water is used to change the concentration of acetic acid. The direct proportions of the components – acetic acid and distilled water are determined by the formula:

$$\frac{\text{Asid}}{\text{Water}} = \frac{Z2}{Y2}, \quad (2)$$

where $Y2$ – water percentage;

$Z2$ – percentage of concentrated acid

- 3) Method for determining the pH value using a pH -meter.

The concentration of acid solution is closely related to such parameter as the pH value [4], which is a measure of the acidity of solutions. The pH parameter itself is a way of expressing the activity of hydrogen cations $C[H^+]$ in solutions and is uniquely related, under equilibrium conditions, to the acid concentration.

The analysis of the presented methods clearly identified the experiment using a pH -meter as basic.

A portable pH -meter is used to study the effect of temperature on the pH value (which often occurs in field conditions), [5]. The type of portable pH -meter used is shown in Fig. 1. The research itself took place at the Lincoln Park Chemical Laboratory, Chicago, USA.



Fig. 1. Portable pH meter

Experimental results. The experiment, carried out at the Lincoln Park laboratory, implements a rapid measurement process and is based on the determination of pH values. pH values indirectly characterize the concentration of acetic acid, and express measurements using a pH -meter allow you to expand the temperature range of CH_3COOH and eliminate the increased value of methodological error in measurement. The hardware error of the pH meter needs to be studied.

The results of determining the pH values of acetic acid at temperatures of 20 °C, 35 °C, 50 °C, 75 °C are summarized in Table 1.

The experiment used a measurement method through multiple observations. The sample of values consisted of five observations, which is sufficient to make a decision.

For the experiment, a solution of acetic acid with a concentration of 1 mol/l was used, obtained by mixing the original acetic acid with a concentration of 6 mol/l (10 ml) and distilled water (50 ml) (2).

Table 1

Results of determining the pH values of CH_3COOH at different temperatures CH_3COOH

Test	$T1, ^\circ C$ (20 °C)	$T2, ^\circ C$ (35 °C)	$T3, ^\circ C$ (50 °C)	$T4, ^\circ C$ (75 °C)
1	2.1	2.1	1.8	1.6
2	2.0	1.8	1.7	1.3
3	2.1	1.9	1.8	1.1
4	2.0	1.9	1.7	1.1
5	2.1	1.8	1.5	1.0

The results are presented in Table 1 look quite densely. But the assessment of the instrumental measurement error requires some statistical processing.

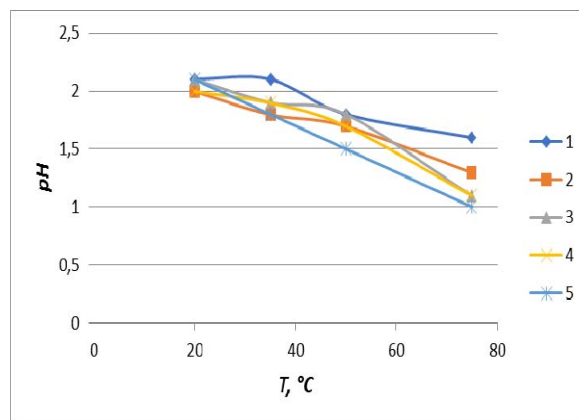
Analysis of experimental results. The obtained experimental results require certain processing. This is necessary to obtain reliable assessments of the results, correct interpretation, drawing conclusions and formulating ways for further research.

Definition of statistical moments of the 1st and 2nd order. Characteristics that demonstrate the behavior of the pH value when the temperature changes in the range of 20 °C – 75 °C are set based on the data in Table 1. Experiments were carried out for five samples with a concentration of $C_K = 1 \text{ mol/l}$.

Based on the data from the same Table 1, a range of characteristics is constructed that demonstrates the behavior of the pH value with temperature changes. The range of characteristics itself is shown in Fig. 2.

The presence of Table 1 data and the range of characteristics (Fig. 1) allows to determine the statistical moments of the 1st and 2nd order – mathematical expectation (M), dispersion (D), standard deviation (SD) [6]. The indicated statistical points are presented in Table 2.

Fig. 3 shows a graphical dependence identifying the change in the mathematical expectation of the pH value as a function of temperature. Fig. 4 shows a graphical dependence identifying the change in the dispersion of the pH value as a function of temperature. Data for graphical dependencies are obtained from Table 2.

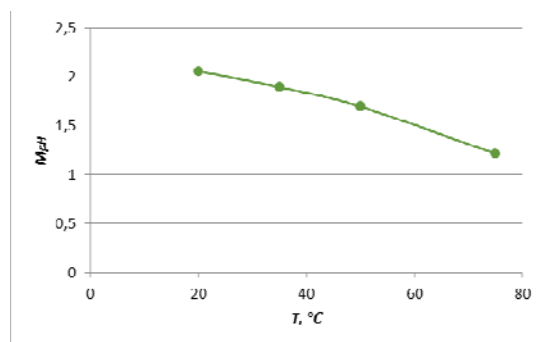
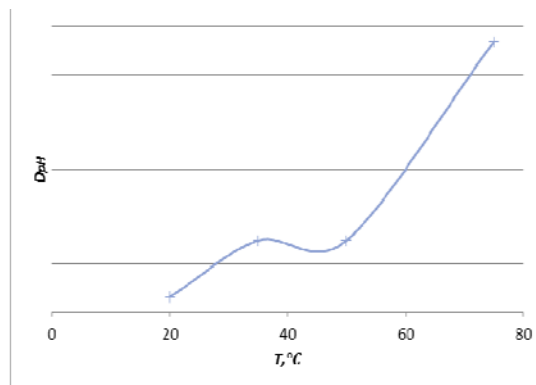
Fig. 2. Range of characteristics $pH = f(T)$:

1 – test 1; 2 – test 2; 3 – test 3;
4 – test 4; 5 – test 5.

Table 2

Table of mathematical expectation, dispersion, standard deviation

T	M	D	SD
20 °C	2.06	0.003	0.012
35 °C	1.90	0.015	0.060
50 °C	1.70	0.015	0.060
75 °C	1.22	0.057	0.228

Fig. 3. Dependence $M_{pH} = f(T, ^\circ C)$ Fig. 4. Dependence $D_{pH} = f(T, ^\circ C)$

According to Fig. 4 it is obvious that the spread of characteristics at a temperature of 75 °C (dispersion and standard deviation data) is an order of magnitude higher than at temperatures of 20 °C, 35 °C, 50 °C. This indicates

either a methodological error (inaccurate measurement) or an instrumental error (the normal operation of a pH meter is limited to a certain temperature range, the pH meter does not work well at high temperatures).

Definition of dissociation parameters. Dissociation parameters are important components when analyzing the behavior of an acid under conditions of temperature change. The main dissociation parameters include [7]:

- degree of dissociation α ;
- dissociation constant K_a .

Conversion of molar concentration to mass concentration is determined from the formula:

$$\omega = \frac{C_M \cdot M}{10 \cdot \rho}, \quad (3)$$

where C_M – molar concentration (1 mol/l) of solution;

ω – mass fraction of acid (%) in solution;

ρ – density of acetic acid solution (g/cm^3). For a solution of acetic acid with $C_M = 1 \text{ mol/l}$ – the density $\rho = 1.0 \text{ g/cm}^3$ (For a solution of acetic acid);

M – molar mass of acetic acid (g/mol).

From (3) the mass fraction of acetic acid in the solution is determined:

$$\omega = \frac{1 \text{ mol/l} \cdot 60 \text{ g/mol}}{10 \cdot 1 \text{ g/cm}^3} = 6 \% .$$

The result obtained determines that the 1 mol/l acetic acid solution used in the research is equivalent to table edible vinegar. Working with such a solution of acetic acid does not require any special precautions.

As for the molar concentration of acetic acid C_M , it determines the concentration of hydrogen cations $C[H^+]$ under equilibrium conditions [8].

The transition from pH number to $C[H^+]$ is carried out according to the formula:

$$pH = -\lg(C[H^+]). \quad (4)$$

From formula (4) it follows:

$$C[H^+] = 10^{-pH}. \quad (5)$$

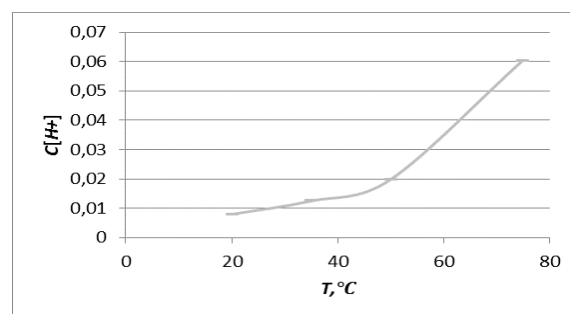
Based on (4) and (5), the Table 3 was formed.

Table 3

The data pH and $C[H^+]$ for temperatures 20°C , 35°C , 50°C , 75°C

$T, ^\circ\text{C}$	20	35	50	75
pH (averaged)	2.09	1.90	1.70	1.22
$C[H^+]$	0.0081	0.0126	0.0199	0.0603

Dependence $C[H^+] = f(T, ^\circ\text{C})$ for the experiment according to the data in Table 3 is shown in Fig. 5.

Fig. 5. Dependence $C[H^+] = f(T, ^\circ\text{C})$

The degree of dissociation α at temperatures of 20°C , 35°C , 50°C , 75°C is determined by the formula:

$$\alpha = \frac{n}{N} = \frac{C[H^+]}{C_M} = \frac{10^{-pH}}{1.0}. \quad (6)$$

As $C_M = 1.0$, then, based on (6):

$$\alpha = C[H^+] = 10^{-pH}. \quad (7)$$

Dissociation constant K_a at temperatures of 20°C , 35°C , 50°C , 75°C is determined by the formula [9]:

$$K_a = \frac{C[H^+] \cdot C[CH_3COO^-]}{C[CH_3COOH]}.$$

This formula, subject to equilibrium, is presented as follows:

$$K_a = \frac{10^{-pH}}{C_M - 10^{-pH}} \quad (8)$$

The calculated data for the degree of dissociation α (7) and dissociation constant K_a (8) are summarized in Table 4.

Table 4

The data α and K_a for the temperatures 20°C , 35°C , 50°C , 75°C

$T, ^\circ\text{C}$	20	35	50	75
α	0.0081	0.0126	0.0199	0.0603
K_a	0.00007	0.00016	0.00041	0.00386

When making calculations to determine the dissociation constant K_a in Table 4, an intermediate table has been formed Table 5.

Table 5

Intermediate table by definition K_a for the temperatures 20°C , 35°C , 50°C , 75°C

$T, ^\circ\text{C}$	20	35	50	75
10^{-2pH}	0.0000	0.0001	0.0004	0.0036
10^{-pH}	0.0081	0.0125	0.0199	0.0602
$C_M - 10^{-pH}$	0.9918	0.9874	0.9800	0.9397

Based on the data in Table 4, graphical dependencies $\alpha = f(T_i)$ (7) and $K_a = f(T_i)$ (8) are built, respectively.

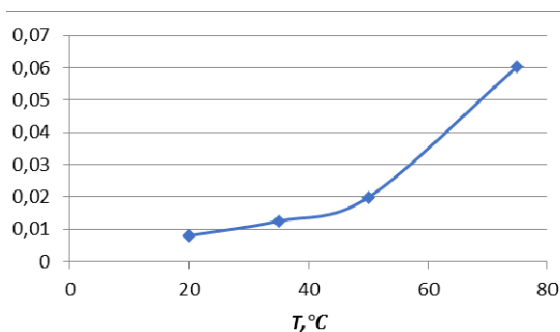


Fig. 6. Dependence $\alpha = f(T_i)$

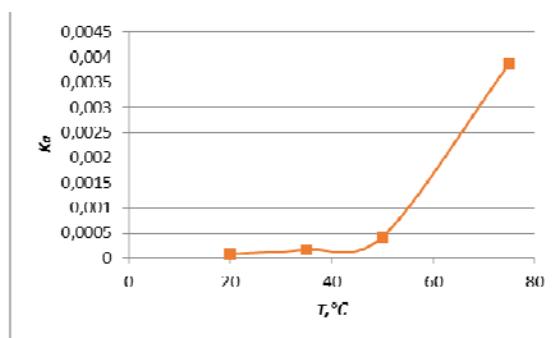


Fig. 7. Dependence $K_a = f(T_i)$

Dependence approximation of the dissociation constant on temperature. The resulting calculated-experimental dependence $K_a = f(T_i)$ is easily approximated by computer means. The approximation itself is shown in Fig. 8.

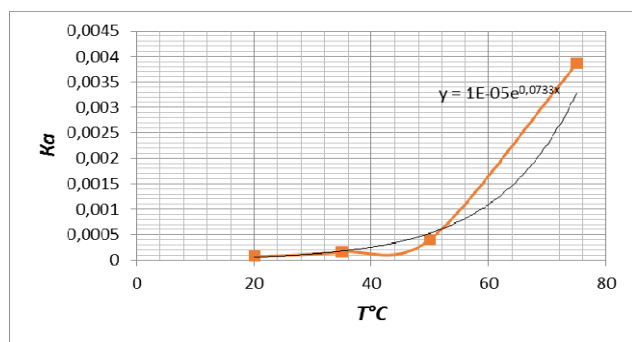


Fig. 8. Dependence approximation $K_a = f(T_i)$

According to the data in Fig. 8, the dissociation constant K_a varies exponentially and can be approximated by the equation:

$$K_a = 1 \cdot 10^{-5} \cdot e^{0.0733 \cdot T, ^\circ C} \quad (9)$$

Based on (9), it is obvious that the change in dissociation parameters (degree of dissociation and dissociation constant K_a) has a clearly expressed nonlinear character and can be described by an exponential dependence. The well-known equation of the

approximating curve makes it possible to carry out circuit-technical temperature correction in the measuring pH meter [10].

Calculation of measurement errors. The true value of the acetic acid concentration is found as the arithmetic mean of the data for all five samples at each specific temperature in the Table 1:

$$C_{KT} = \frac{C_{K1} + C_{K2} + C_{K3} + C_{K4} + C_{K5}}{5} \quad (10)$$

Temperatures of Table 1 are nodal points for calculating errors.

The results of calculations using formula (10) are summarized in Table 6 and reflect the true values of the measurement results. For the experiment, the parameter pH to be considered the true measurement result.

To determine the error in the result of measurements and according to measurements theory, it is necessary to determine the maximum random measurement error $t\sigma_{C_K}$ [11]. Then the measurements result is written in the form:

$$C_K = C_{KT} \pm t\sigma_{C_K}, \quad (11)$$

where t – Student's t-test, which is determined from normative tables based on confidence probability P_D and number of observations n ;

σ_{C_K} – standard deviation of the measurement result.

The standard deviation of the measurement result is determined based on the expression:

$$\sigma_{C_K} = \frac{\sigma}{\sqrt{n}}, \quad (12)$$

where σ – standard deviation of a number of observations;

$n = 5$ – number of observations in the experiment.

The standard deviation of a number of observations σ is determined by the formula:

$$\sigma = \pm \sqrt{\frac{\sum \rho^2}{n-1}}, \quad (13)$$

where ρ – residual observation error, equal to the difference between the observation result and the measurement result:

$$\rho = C_{ki} - C_{KT} \quad (14)$$

Data on the residual error of the experiment at nodal temperature points are also summarized in Table 6.

The initial data for calculating errors are the data in Table 6.

Table 6

Data pH, σ , σ_{pH} of acetic acid at different temperatures

	20°C	35°C	50°C	75°C
pH	2.06	1.90	1.70	1.22
σ	0.0548	0.1225	0.1225	0.2387
σ_{pH}	0.0245	0.1104	0.1104	0.2151

Based on the data in Table 6 and expressions (11) – (14), measuring results of the parameter at nodal temperature points are written as follows:

- at $T = 20^{\circ}\text{C}$ $C_K = 2.06 \pm 0.05$;
- at $T = 35^{\circ}\text{C}$ $C_K = 1.90 \pm 0.11$;
- at $T = 50^{\circ}\text{C}$ $C_K = 1.70 \pm 0.11$;
- at $T = 75^{\circ}\text{C}$ $C_K = 1.22 \pm 0.21$.

Conclusions. The rapid method selected for experimentation in the study relies on a technique that has been carefully reviewed in these researches. The technique that implements the rapid method involves recalculating the pH value into the real concentration of acetic acid C_M according to formula (5) using the equilibrium condition.

The presented method implements indirect measurements, which is not very good from the point of view of reducing measurement errors. However, indirect measurements, which are carried out in this case, are automated by a pH meter with minimal time expenditure and minimal subjective destabilizing factors introduced by the operator.

Other important conclusions from the studies are:

1. The change in dissociation parameters (degree of dissociation and dissociation constant K_a) has a clearly expressed nonlinear character and can be described by an exponential dependence.
2. The values of the parameters (degree of dissociation α and dissociation constant K_a) increase with increasing temperature for a solution of acetic acid CH_3COOH .
3. Based on the behavior of the dissociation constant K_a , it can be argued that with increasing temperature, the acid CH_3COOH becomes more aggressive (strong).

Prospects for further research. The preferable use of a portable pH -meter to determine the effect of temperature on the concentration of acetic acid was clearly demonstrated by the studies, which is shown in the conclusions. However, the obtained experimental results and the corresponding statistical processing of the results determined further approaches to improving pH -meters.

During the experiment, it was determined that the spread of characteristics at a temperature of 75°C (dispersion and standard deviation data) is an order of magnitude higher than at temperatures of 20°C , 35°C , 50°C . This indicates either a methodological error (inaccurate measurement) or an instrumental error (the normal operation of a pH meter is limited to a certain temperature range; the pH meter does not work well at high temperatures).

Elimination of the specified instrumental error of the device (expanding the temperature range of the pH -meter without losing the accuracy class of the device) is possible using the following approaches [12]:

- linearization of characteristics;
- piecewise linear approximation;
- introduction of additive amendments due to the use of a Table with corresponding amendments;
- end-to-end approximation of the characteristic.

Each of the presented methods has its own advantages and disadvantages. Each of the presented methods has found wide application in practice.

The presence of a microprocessor unit, as well as hardware and software in a portable pH -meter, is the most appropriate approach to compensate for temperature errors and, accordingly, expand the temperature range of the normal operation of the device and to use the compilation of correction tables. The resulting correction tables are easily entered into the ROM memory [13] of the device. Such recommendations will somewhat reduce the time parameters of the measurement process, but will significantly expand the technical and accuracy parameters of the pH -meter.

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Надійшла (received) 25.09.2023