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STUDY OF THE METROLOGICAL CHARACTERISTICS OF A FLOW-TYPE MILK ACIDITY ANALYZER FOR MILKING EQUIPMENT

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The paper presents the results of a study on the metrological characteristics of a flow-type milk acidity analyzer designed for integration into milking equipment to enable continuous real-time monitoring of raw milk quality. The relevance of this research is driven by the needs of modern livestock farming for the implementation of digital solutions within the framework of the Smart Farming concept. Milk acidity is a key indicator reflecting the microbiological status, freshness, and suitability of milk for processing, and its timely control allows for the prompt detection of hygienic violations during milking or early signs of animal diseases.

The proposed design of the flow analyzer is based on the ionometric method for measuring pH using a glass pH-selective electrode, a reference electrode, and a DS18B20 digital temperature sensor. The measuring unit is built on an ATMega16 microcontroller, integrated with a TL082 operational amplifier, which ensures high-precision measurement of the electrode system potentials. Signal processing and measurement control are implemented via PC software, which also provides electrode calibration, acidity calculation in pH and °T units, and digital and graphical visualization of results.

To determine the metrological characteristics of the analyzer, comparative measurements were carried out using a laboratory-grade pH meter S20 (Mettler Toledo) in buffer solutions with known pH values. It was established that the electrode response is linear with a slope of 59.62 mV/pH (theoretical value – 59.16 mV/pH), the measurement range is 1.68–9.18 pH, the signal dispersion is 3.00 mV², resolution – 0.01 pH, and the signal stabilization time ‒ up to 25 seconds. The average deviation of the results does not exceed ±0.03 pH, which confirms compliance with the requirements for operational acidity monitoring in flow conditions.

The developed analyzer is competitive in terms of key metrological parameters and has strong potential for industrial application. Further research will focus on the implementation of automatic interpretation algorithms based on processing standards, as well as adaptation of the device to farms of various scales.

Key words: *milk, milk acidity, milking equipment, technical condition, pH meter, ionometric method, flow analyzer, metrological characteristics, quality control, automation, measurement.*

Eq. 5. Fig. 4. Table. 3. Ref. 15.

1. Problem formulation

Modern livestock farming is increasingly focused on implementing high-tech means of automation and digitalization in the processes of monitoring the quality of raw milk. One of the key indicators of milk quality is its acidity, which serves as an important marker of microbiological stability, freshness, and suitability for further processing [1, 2]. Changes in acidity may result from sanitary violations in milking equipment, the onset of product spoilage, or the presence of diseases in animals, particularly subclinical mastitis [3].

In traditional practice, milk acidity control is carried out using laboratory methods that require sample collection and a certain analysis time. This approach does not allow for prompt response to changes in raw milk quality directly during milking. In modern dairy farming, especially on farms with robotic milking or flow milk collection systems, this creates risks of losses and decreases overall production efficiency.

To implement the Smart Farming concept, it is necessary to introduce flow analyzers of acidity integrated directly into milking equipment. Such systems enable real-time monitoring of milk quality indicators, detection of pathological changes or microbiological spoilage, as well as automation of the milk selection or rejection process [4, 5].

Recent studies confirm the feasibility of accurate measurement of milk acidity or pH in flow using optical, electrochemical, or spectroscopic sensors that can be embedded in milking units [6, 7]. Furthermore, the use of machine learning combined with in-line NIR analysis significantly improves the accuracy and stability of results even under varying milking conditions [8].

At the same time, practical implementation of such systems requires metrological justification—determining parameters of accuracy, sensitivity, reproducibility, stability, and measurement reliability. This is critically important for the further standardization of devices, their certification, and integration into milking equipment.

Thus, the development and determination of metrological characteristics of a flow milk acidity analyzer, capable of operating as part of milking equipment, is a relevant scientific and practical task that will contribute to improving milk quality, the efficiency of the technological process, and the introduction of innovations in livestock farming.

2. Analysis of recent research and publications

Several methods are used to determine milk acidity, the main ones being titration, conductometric, and pH-metric methods [9–12]. A comparative characterization of these methods is presented in Table 1.

Analysis of the acidity determination methods shown in Table 1 indicates that the ionometric method is optimal for monitoring the acidity of milk and dairy products. Its advantages include relatively simple equipment design, measurement duration of a few seconds, and the possibility of conducting measurements both in samples and in flow. The main drawback of the ionometric method is the need for temperature compensation when the milk temperature differs from 20 °C to ensure high measurement accuracy.

Table 1

Comparative characteristics of milk acidity determination methods

Method	Principle	Characteristics
Titration	Quantitative analysis of acid in milk by neutralization with alkaline solution to equivalence point	Complex measurement equipment design; measurement duration of several minutes; unsuitable for continuous acidity monitoring (e.g., in flow)
Conductometric	Determination of acid concentration in milk by its electrical conductivity	Relatively simple measuring circuit and sensor design; capability to simultaneously monitor acidity and temperature with one device. The error of acidity determination of fresh milk by this method is up to ±3 °T, which does not meet technological requirements in the dairy industry; seasonal changes in average milk conductivity significantly affect measurement results.
pH-metric	Determination of milk acidity by pH value	Measurement duration from a few seconds to several tens of seconds; relatively simple measuring equipment design; insignificant dependence of pH value on the content of proteins, phosphates, citrates, etc. in milk; applicable for both periodic and continuous acidity monitoring. To ensure acidity measurement accuracy that meets technological requirements, temperature compensation is necessary.

The pH value of milk undergoing lactic acid fermentation is a function of several variables [13–15]:

$$pH_M = f\left(pH_0, \beta, \alpha, t\right), \tag{1}$$

where pH_0 is the pH of fresh milk; α is the content of lactic acid in the milk; β is the coefficient characterizing the buffering properties of milk; t is the temperature of the milk.

In turn, the values of pH_0 and β depend on the content of proteins and salts of phosphoric and citric acids in the milk. Thus, only the parameter α in expression (1) characterizes the influence of lactic acid content on the pH of milk. That is, the pH of milk is not an unambiguous indicator of lactic acid concentration in milk because milk is a complex polydisperse buffered medium. However, the influence of proteins and salts on the pH value does not cause significant error in practice, and if the effect of temperature on acidity is compensated, then the pH measurement method meets the specified technological requirements for monitoring the acidity of bulk milk.

The averaged relationship between the pH value and the titratable acidity A of bulk milk at 20 °C can be expressed as follows [13–15]:

$$pH = pH_{0avg} - \frac{h_0}{K_T}\left(A - A_0\right), \text{ або } pH = 6,69 - 0,056 \cdot \left(A - 17\right), \quad (2)$$

where h_0 is the regression coefficient, h_0 = 6.2 pH/% titratable acidity; K_T is the coefficient, K_T = 111°T/% titratable acidity; A_0 is the titratable acidity of fresh milk, A_0 = 17 °T; pH_{0avg} is the average pH value of fresh milk, pH_{0avg} = 6.69 pH.

In practice, the ionometric method for determining acidity is implemented as follows. A pH-selective measuring electrode (an electrode whose potential changes depending on the activity of hydrogen ions in the solution) and a reference electrode, whose potential is considered constant under given conditions, are immersed into the analyzed solution (milk). Then, using a measuring device (pH meter), the difference in electrode potentials is determined, which is a function of the acidity of the solution [13–15]. Specifically, if a glass electrode is used as the measuring electrode, this function is linear and described by the Nikolsky equation [13–15]:

$$E = E' + \frac{RT}{F} \cdot pH, \quad (3)$$

where E is the electrode system potential, mV; E′ is the standard potential (electrode system potential at pH = 0), mV; R is the universal gas constant, J/(mol·K); T is the temperature, K; F is the Faraday constant, C/mol.

3. The purpose of the article

The aim of this work is to investigate the metrological characteristics of a flow acidity analyzer for milk, designed for integration into milking equipment, to ensure reliable, prompt, and continuous monitoring of acidity during the milking process.

4. Results and discussion

Let us define the basic technical requirements that must be met for a flow milk acidity analyzer using a glass electrode:

1. The potential difference between the glass electrode and the reference electrode, according to equation (3), is determined by the pH value and the temperature of the solution in which these electrodes are immersed. The potential of the glass electrode can reach several hundred millivolts.
2. The glass electrode has a very high output resistance (ranging from tens to several hundreds of megaohms and even higher). Therefore, measuring the potential difference between the glass electrode and the reference electrode requires measuring devices with an input resistance of about 10^{12} ohms [5]. Such a high input resistance can be ensured by using operational amplifiers in the measuring circuit.
3. The time required for the potentials to stabilize to equilibrium ranges from several seconds to several tens of seconds. Therefore, high-frequency interferences can be filtered out.
4. The pH value significantly depends on the temperature of the solution; hence, to improve the accuracy of pH determination, measurement of the solution temperature is mandatory.
5. Characteristics of glass electrodes (such as the slope of the electrode characteristic and the position of the isopotential point) change over time depending on storage and usage conditions. Therefore, to achieve acceptable accuracy in pH measurements, effective and rapid calibration methods must be provided.

Considering these conditions, the proposed design of the laboratory flow milk acidity analyzer and its prototype (Fig. 1) were created, consisting of the following components:

– an electrode system comprising a glass pH-selective electrode and a reference electrode;

– a temperature sensor;

– a measuring unit, which measures the potentials of the electrodes and temperature sensor, converts them into digital form, and exchanges data with a personal computer;

– a personal computer for registering and processing signals sent by the measuring unit and controlling the measurement process.

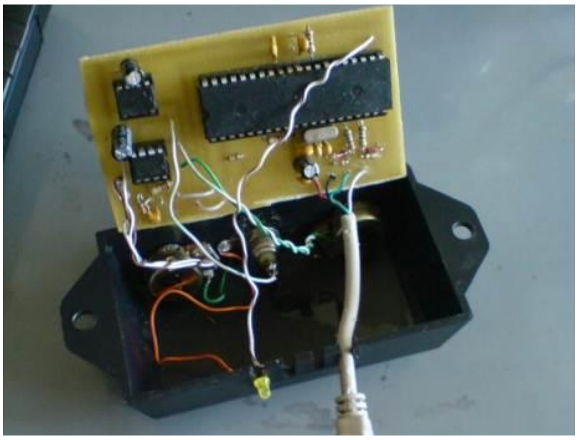


Fig. 1. Measuring Unit of the Flow Milk Acidity Analyzer

The main component of the measuring unit is the ATMega16 microcontroller with a built-in multi-channel analog-to-digital converter (ADC). Signals from the electrodes and the temperature sensor are fed to the corresponding microcontroller inputs; these signals are converted into digital form by the ADC and transmitted via a USB interface to the computer for further processing and visualization. The USB protocol is implemented in software, without the use of additional specialized chips, which significantly simplifies the circuit design and reduces the overall device cost.

To amplify the signal from the electrode system and to meet the requirement for high input impedance of the measuring device, a dual operational amplifier based on the TL082 chip was additionally included in the input stage of the measuring unit. This amplifier, on the one hand, has an input impedance of about $1 \cdot 10^{12}$ ohms and, on the other hand, is characterized by low cost.

The entire assembled flow milk acidity analyzer is shown in Fig. 2. For the device implementation, a laboratory combined pH-selective electrode ESC-10605 was used, which consists of a glass measuring electrode and a reference electrode housed in a single body, as well as a DS18B20 temperature sensor.

Fig. 2. Study of Metrological Characteristics of the Flow Milk Acidity Analyzer

The software of the developed flow milk acidity analyzer is divided into microcontroller software and personal computer software. The computer software is designed to perform the following functions:

– configuration and start of the ADC;

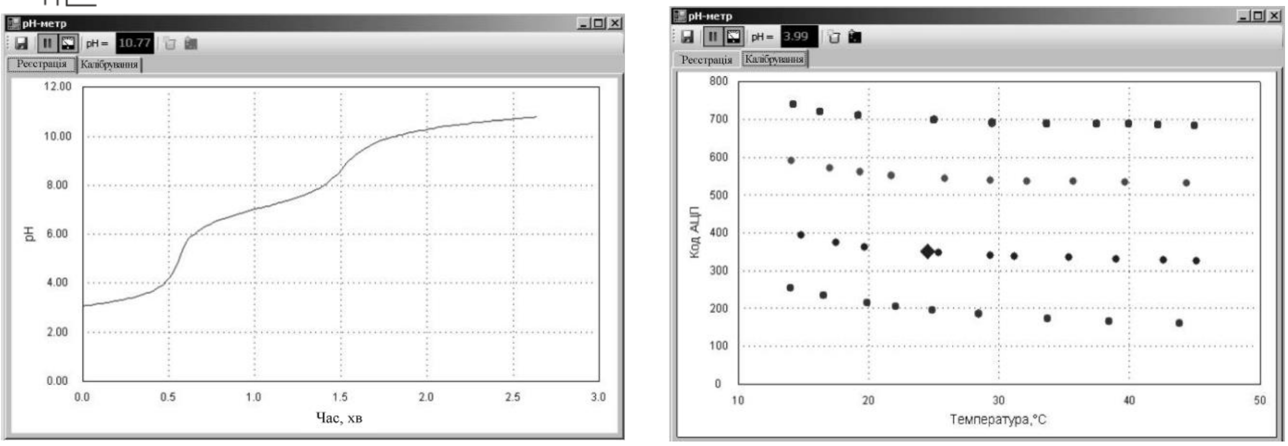
– verification of connection of the measuring electrode and temperature sensor;

– reading data and interpreting them as electrode potential in millivolts and temperature in °C;

– calculation of the acidity of the analyzed solution in pH units and, upon operator command, conversion of the obtained values into titratable acidity according to equation (2);

– storage and/or visualization of the measured acidity values in numerical and graphical forms (Fig. 3a);

– calibration of the electrode system (Fig. 3b).



a ***b***

Fig. 3. Display of measurement results (a) and calibration of the electrode system (b) conducted using the flow milk acidity analyzer

To verify the metrological performance of the developed flow analyzer, parallel measurements of the pH of buffer solutions were conducted using this device and the laboratory pH meter S20 from Mettler Toledo, the main parameters of which are listed in Table 2.

Table 2

Metrological parameters of the S20 pH meter

Measured Quantity	Parameter	Value
pH	Measurement range	0…14
	Resolution	0.01
	Measurement accuracy	± 0.01
Electrode potential, mV	Measurement range	–1999…1999
	Resolution	1
	Measurement accuracy	± 1
Temperature, °C	Measurement range	–5…105
	Resolution	0.1

Measurements were conducted in the following sequence:

– Prior to measurements, the combined electrode was kept in distilled water for 24 hours;

– The electrode and temperature sensor were connected to the measuring unit of the flow analyzer;

– The electrode and temperature sensor were immersed in a container holding 30 ml of solution with pH = 9.18. After stabilization of the electrode potential, the result was recorded;

– The electrode and temperature sensor were rinsed with distilled water;

– Measurements were sequentially performed in solutions with pH values of 6.86, 4.01, and 1.68. Each measurement was repeated three times in each solution;

– The electrode and temperature sensor were connected to the S20 pH meter and the above measurements were repeated again.

The measurement results obtained using both devices are presented in Table 3 and in Figure 4. The time required to reach a stable electrode potential when using both devices was 10 to 25 seconds, which is typical for such measurement systems.

Statistical processing of the measurement results showed that in both cases the dependence of the electrode system potential on the pH value is linear and is described by the equations:

– for measurements using the flow acidity analyzer:

$$E = 126{,}38 - 57{,}52 \cdot pH, \qquad (4)$$

– for measurements using the pH meter:

$$E = 55{,}86 - 59{,}22 \cdot pH. \qquad (5)$$

Table 3

Measurement results of electrode potentials in solutions with different pH values

Measuring Device	Measurement №	Solution Acidity, pH units			
		1.68	4.01	6.86	9.18
		Electrode Potential, mV			
Flow Milk Acidity Analyzer	1	29	– 101	– 270	– 400
	2	27	– 104	– 270	– 404
	3	30	– 102	– 267	– 401
pH Meter S20	1	– 44	– 182	– 349	– 487
	2	– 45	– 182	– 349	– 489
	3	– 42	– 181	– 349	– 489

The discrepancy in the constant terms in equations (4) and (5) does not affect the accuracy of pH determination, since this parameter is compensated during measurements. The obtained slope values in both equations are close to the theoretical value of 59.16 mV/pH.

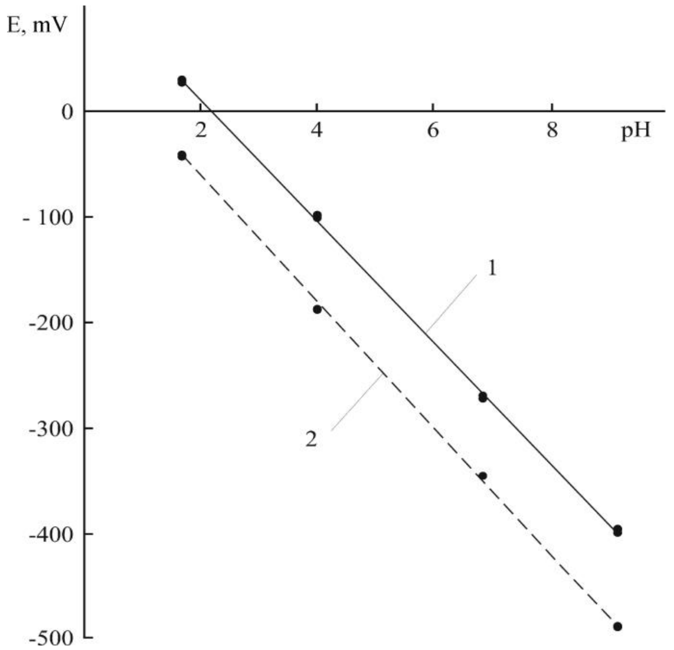


Fig. 4. Calibration characteristics of the electrode system obtained using the flow milk acidity analyzer and the reference device: 1 – data from the flow milk acidity analyzer; 2 – data from the pH meter S20

The variance of measurements conducted using the flow acidity analyzer is $s_0^2 = 3.00$ mV², while for the pH meter it is $s_0^2 = 1.42$ mV².

5. Conclusion

A prototype of a flow acidity analyzer for milk intended for dairy milking equipment has been developed and created. The measurement results obtained using this device indicate that the metrological characteristics of this flow analyzer are comparable to those of existing pH meters. The next step is to improve the analyzer's software to implement the function of assessing the milk's suitability for processing based on its acidity in the flow.

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ДОСЛІДЖЕННЯ МЕТРОЛОГІЧНИХ ХАРАКТЕРИСТИК ПОТОКОВОГО АНАЛІЗАТОРА КИСЛОТНОСТІ МОЛОКА ДЛЯ МОЛОЧНО-ДОЇЛЬНОГО ОБЛАДНАННЯ

У роботі представлено результати дослідження метрологічних характеристик потокового аналізатора кислотності молока, призначеного для інтеграції у молочно-доїльне обладнання з метою забезпечення безперервного моніторингу якості молочної сировини в реальному часі. Актуальність дослідження зумовлена потребами сучасного тваринництва у впровадженні цифрових рішень для реалізації концепції «розумної ферми». Кислотність молока є ключовим показником, що відображає *мікробіологічний стан, свіжість і придатність молока до переробки, а її своєчасний контроль дозволяє оперативно виявляти порушення гігієнічних умов доїння або початкові ознаки захворювань у тварин.*

Запропонована конструкція потокового аналізатора базується на іонометричному методі вимірювання pH із використанням скляного pH-селективного електрода, електрода порівняння та цифрового температурного датчика DS18B20. Вимірювальний блок побудовано на мікроконтролері ATMega16, у схему якого інтегровано операційний підсилювач TL082, що забезпечує високоточне вимірювання потенціалів електродної системи. Обробка сигналів та управління процесом вимірювання реалізовані через програмне забезпечення для ПК, яке також забезпечує калібрування електродів, розрахунок кислотності в одиницях pH і °T, а також візуалізацію результатів у цифровій і графічній формах.

Для визначення метрологічних характеристик аналізатора проведено порівняльні вимірювання з лабораторним pH-метром S20 (Mettler Toledo) у буферних розчинах із відомими значеннями pH. Встановлено, що електродна характеристика має лінійний характер із кутом нахилу 59,62 мВ/pH (теоретичне значення – 59,16 мВ/pH), діапазон вимірювання – 1,68…9,18 pH, дисперсія результатів – 3,00 мВ², роздільна здатність – 0,01 pH, час встановлення стабільного сигналу – до 25 с. Середнє відхилення результатів не перевищує ±0,03 pH, що підтверджує відповідність вимогам до оперативного моніторингу кислотності в потоці.

Розроблений аналізатор є конкурентоспроможним за основними метрологічними характеристиками і має потенціал для промислового застосування. У подальших дослідженнях планується реалізація алгоритмів автоматичної інтерпретації результатів з урахуванням норм переробки, а також адаптація приладу до умов фермерських господарств різного масштабу.

Ключові слова: *молоко, кислотність молочно-доїльне обладнання, технічний стан, pH-метр, іонометричний метод, потоковий аналізатор, метрологічні характеристики, контроль якості, автоматизація, вимірювання.*

Ф. 5. Рис. 4. Табл. 3. Літ. 15.

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