



Section 2. Chemistry

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DEVELOPMENT OF A SEMICONDUCTOR-BASED SELECTIVE SENSOR FOR ACETONE VAPOR MONITORING AND DETERMINATION OF ITS METROLOGICAL CHARACTERISTICS

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Abstract

Based on the results of the conducted studies, a method for ensuring the selectivity of acetone detection has been proposed using semiconductor sensors integrated into a single unit containing catalysts with sufficient activity toward the analyzed mixture. The absolute and relative errors and variation of the analytical signal were determined in accordance with the parameters specified by the State Standard GOST 13320–81 and were found not to exceed the established limits. At a confidence level of 0.95, the total reduced error over the measured concentration range fully complies with the requirements of the State Standard GOST 52033–2003 for instruments of this type and does not exceed $\pm 1.5\%$.

Keywords: acetone, semiconductor sensor, catalyst, sensitivity, selectivity, dynamic parameter, absolute and relative errors, variation

Introduction

Acetone is a highly flammable substance, and its mixture with air is explosive (**GOST 2768–84**). In addition, acetone is a psychoactive substance that can adversely affect the human central nervous system (**GOST 12.1.007–76**). Therefore, highly sensitive and rapid methods for monitoring the concentration of acetone in the air are required to ensure occupational safety at various industrial and other facilities (**GOST 32419**). The problem of acetone monitoring can be

addressed through the development of reliable sensors and gas analyzers that meet all the required metrological characteristics. Semiconductor sensors and gas analyzers are among the most promising and reliable instruments and devices for monitoring combustible components in gas mixtures, particularly acetone.

Experimental Results and Discussion

Based on the experimental results obtained, a method was developed to ensure

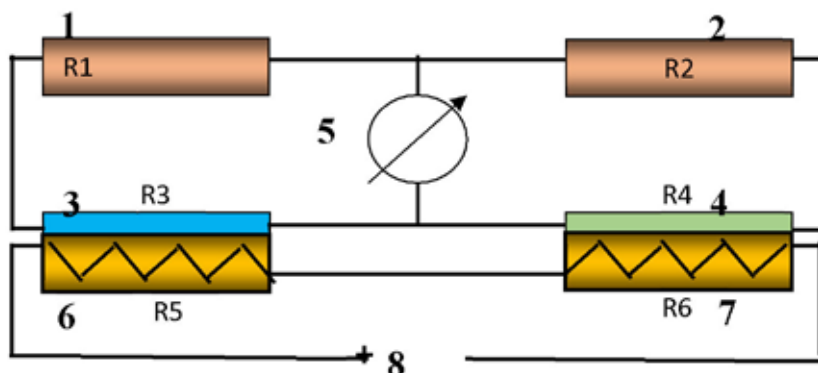
the selectivity of a semiconductor sensor for acetone in the presence of CO, H₂, and hydrocarbons. The proposed method is based on the use of sensing elements containing catalysts with different levels of activity toward the components of the gas mixture.

Using the developed catalysts, selective semiconductor acetone sensors were fabricated. Each sensor consisted of two sensing

elements – a measuring element and a compensation element – and two resistors connected in a bridge circuit (Fig. 1).

The gas-sensitive film of the measuring element had the composition **SiO₂/ (1.0TiO₂ + 0.75CuO)**, whereas the composition of the compensation element was **SiO₂/ (0.25Fe₂O₃ + 0Ni₂O₃)**.

Figure 1. Circuit diagram of the sensor for detecting acetone in a gas mixture



1 and 2 – fixed resistors; 3 and 4 – gas-sensitive elements; 6 and 7 – heating elements for the gas-sensitive layer (platinum wires coated with glass); 5 – signal measuring instrument; 8 – current source supplying power to the platinum-wire heaters.

The developed sensors enable the monitoring of micro- and macro-concentrations of acetone in gas mixtures. When the gas mixture enters the sensor chamber, the measuring element of the developed sensor generates a response to CO, H₂, and acetone, whereas the compensation element produces a corresponding response to CO. Under the selected operating conditions, hydrocarbons are practically not oxidized on either sensing element. Consequently, the difference between the signals of the measuring and compensation elements corresponds to the concentration of acetone in the gas mixture.

In accordance with the occupational safety requirements for handling acetone, the following main characteristics of the acetone sensor were determined:

a) dependence of the sensor response on the temperature of the sensing element (i.e., on the magnitude of the voltage/current supplied to the heating layer of the sensing element);

b) dynamic response of the sensor signal;

c) selectivity of the sensor toward the target component (discrimination capability);

d) variation of the output signal over a specified time interval, i.e., the change in the sensor response corresponding to a constant acetone concentration.

The optimal sensor supply voltage was experimentally determined. Its dynamic and calibration characteristics, selectivity, and signal stability were investigated. A batch of five selective semiconductor sensors operating as part of the acetone gas analyzer system was used in the tests.

Experiments to evaluate the semiconductor acetone sensor were conducted under normal, optimized, permissible extreme, and operating conditions. All experiments were performed using instruments and equipment that had undergone metrological certification.

The temperature of the gas-sensitive layer is maintained by the current supplied to the heating element. Therefore, during the development of various semiconductor sensors, the effect of the applied voltage on the sensor response is investigated, and the optimal supply voltage is determined.

The dependence of the response magnitude of the semiconductor acetone sensor

(SAS-Ac) on the voltage applied to the sensor was studied over the voltage range of 1.0–3.5 V. Experiments to determine the optimal supply voltage were conducted under normal operating conditions using

a gas–air mixture containing acetone at a concentration of 100 mg/m³. The results obtained for selecting the optimal supply voltage for the acetone sensor are presented in Table 1.

Table 1. *Dependence of the SAS-Ac response on the voltage applied to the sensor (acetone concentration: 100 mg/m³, n = 5, P = 95%)*

Sensor supply voltage, V	Sensor response, mV		
	$\bar{x} \pm \Delta X$	S	Sr*10 ²
1,0	0,4±0,01	0,01	2,0
1,2	0,5±0,01	0,01	1,4
1,4	0,6±0,01	0,01	1,5
1,6	3,8±0,02	0,02	0,4
1,8	7,6±0,11	0,09	1,2
2,0	8,3±0,16	0,13	1,9
2,2	8,5±0,18	0,15	1,7
2,4	8,4±0,18	0,14	1,6
2,6	8,3±0,16	0,13	1,8
2,8	8,2±0,11	0,08	0,9
3,0	8,9±0,12	0,10	1,2
3,5	7,9±0,14	0,12	1,4

The data presented in (Table 1) show that the optimal supply voltage for the semiconductor acetone sensor (SAS-Ac) is **2.2 V**. An increase or decrease in the supply voltage from the optimal value leads to a decrease in the useful analytical signal and a deterioration in the selectivity of acetone detection.

Considering the intended applications of the sensor and the high toxicity of acetone, an essential requirement for its operation is a rapid response, i.e., a short response time. Therefore, the acetone sensor should have good dynamic characteristics.

The transient response time of the SAS-Ac was investigated under conditions of a stepwise change in the acetone concentration at the sensor inlet. Gas mixtures containing acetone at concentrations of **75 mg/m³ and 150 mg/m³** were used in the experiments. The characteristics of the acetone sensor were evaluated by continuously recording the transient response on the chart tape of a **KSP-4 recording instrument**.

The change in acetone concentration at the inlet of the SAS-Ac was also recorded on

the chart tape and was taken as the starting point for time measurement.

The investigation of the dynamic characteristics of the developed acetone sensor included the study of its time-dependent characteristics, particularly the transient response processes.

Before starting the tests, the sensor baseline (background) signal had to be established. For this purpose, purified argon (or purified nitrogen) was supplied for 10 min to five semiconductor acetone sensors connected in series. The sensor signals, corresponding to the resistance values of the gas-sensitive layers, were recorded using a V7–35 voltmeter.

The transient response time of the SAS-Ac was investigated under conditions of a stepwise change in acetone concentration at the sensor inlet. Gas mixtures containing acetone at concentrations of **75 mg/m³ and 150 mg/m³** were used in the experiments. The characteristics of the acetone sensor were evaluated by continuously recording the transient response on the chart tape of a **KSP-4 recording instrument**.

The change in acetone concentration at the inlet of the SAS-Ac was simultaneously recorded on the chart tape and was taken as the zero point for measuring the response time.

The experiments were conducted under normal conditions: a temperature of **20 °C**, an ambient pressure of **730 mmHg**, and a relative humidity of **60%**.

In the experiments, each test point within the measurement range was characterized by six measurements: three measurements in the forward direction, corresponding to the time required to reach the maximum signal value, and three measurements in the reverse direction, corresponding to the time required to return to the initial signal value.

After the analytical signal of the sensors reached a constant value, the following parameters were determined as the dynamic characteristics of the semiconductor acetone sensor:

to.1 – initial response time, i.e., the time required for the signal to reach 10% of its final value, s;

to.65 – characteristic time, i.e., the time required for the signal to reach 65% of its final value, s;

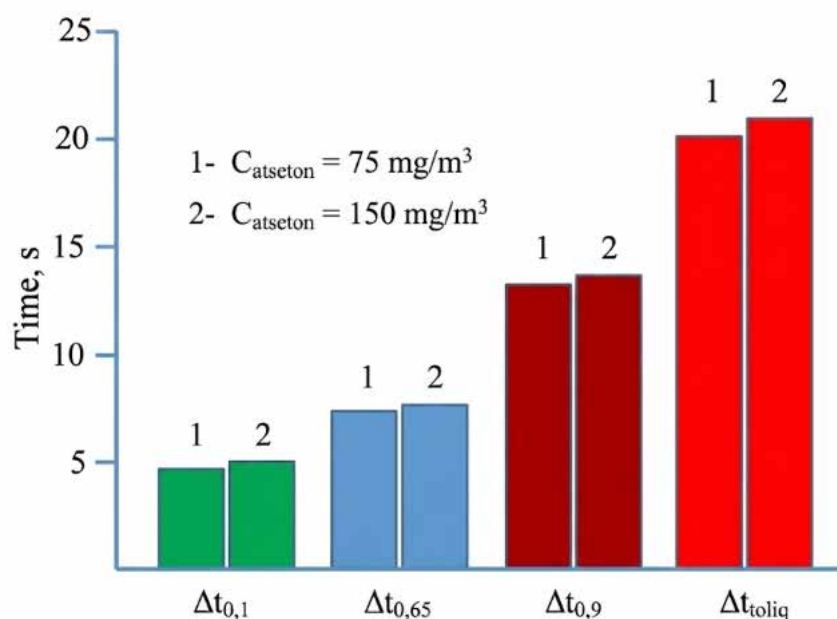
to.9 – signal stabilization time, i.e., the time required for the signal to reach 90% of its final value, s;

t(full) – total measurement time, i.e., the time required for the signal to reach its maximum value, s.

The results of determining the dynamic characteristics of the SAS-Ac are presented in (Fig. 1). As can be seen from the figure, for the gas-sensitive film with the composition **SiO₂/(TiO₂ + 0.75CuO)**, the dynamic parameters of the sensor were as follows:

- **to.1** – initial response time: **5 s**;
- **to.65** – characteristic time: **8 s**;
- **to.9** – signal stabilization time: **13–14 s**;
- **t(full)** – total measurement time: **20–21 s**.

Figure 2. Dynamic characteristics of the semiconductor sensor for acetone detection

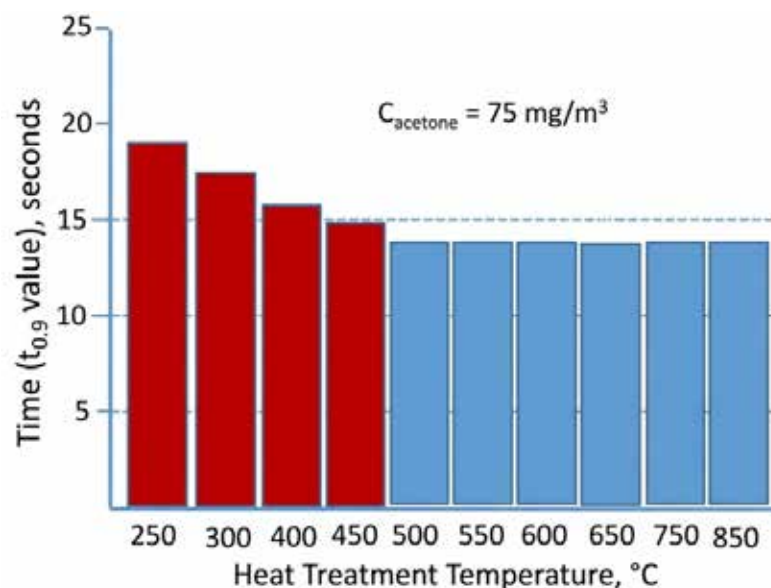


The figure shows that, for the gas-sensitive film with the composition **SiO₂/(TiO₂ + 0.75CuO)**, an increase in the acetone concentration in the gas mixture from **75 mg/m³ to 150 mg/m³** results in a slight increase in the dynamic characteristics of the sensor. In particular, the **total measurement time (t(full))** increases from **20 to 21 s**.

It has generally been established that, for semiconductor sensors, the time required to reach the maximum signal value depends on the heat-treatment conditions of the gas-sensitive film. For the acetone sensor, this parameter was investigated within the temperature range of **250–850 °C**. The dependence of the signal of the **SAS-Ac based on**

SiO₂/(TiO₂ + 0.75 CuO) on the firing temperature of the gas-sensitive material (GSM) is presented in (**Fig. 3**).

Figure 3. Dependence of the signal stabilization time ($t_{0.9}$) on the heat-treatment temperature of the gas-sensitive film during acetone detection ($C_{\text{acetone}} = 75 \text{ mg/m}^3$)



The figure shows that the time required for the sensor signal to reach its maximum value depends on the firing temperature of the gas-sensitive material (GSM). This time decreases with increasing temperature within the range of **250–500 °C** and remains practically unchanged at temperatures above **500 °C**.

The minimum time required for the sensor based on **SiO₂/(TiO₂ + 0.75CuO)** to reach its maximum signal value was **14 s**, corresponding to the signal stabilization time ($t_{0.9}$) of the gas-sensitive material thermally treated at **550 °C**. Considering the dependence of sensitivity on the porosity of the gas-sensitive material, it can be assumed that the gas-sensitive material reaches its maximum porosity at approximately **500 °C**.

The data presented in (**Figure 2 and 3**) confirm that the developed sensors are capable of rapid detection of acetone.

The reproducibility of the sensor response corresponding to the same constant concentration in repeated analyses was investigated using a gas mixture containing **100 mg/m³ of acetone**. Five replicate determinations were performed using five sensors simultaneously.

In these studies, the sensor response was recorded using a self-recording potentiometer. The results of the study demonstrated that the developed sensor, consisting of two

sensing elements, is characterized not only by high selectivity but also by good reproducibility of the analytical signal.

One of the most important characteristics of gas-analytical instruments is the selective determination of a target component in a mixture of analyzed gases. In the semiconductor sensors developed in this study, selectivity was ensured by selecting optimal operating temperatures and appropriate catalysts. The selectivity of acetone detection using the developed sensors was investigated with certified gas mixtures in accordance with the GOST requirements for gas-analytical instruments intended for closed environmental systems and chemical industry facilities.

The selectivity of the semiconductor acetone sensor (SAS-Ac) was determined at a temperature of **350 °C** using standard gas mixtures in the presence of hydrogen, carbon monoxide, and methane. Mixture No. 1 (**acetone + air**) was supplied to the SAS-Ac for **5 min**, and the sensor response was recorded using a digital voltmeter. Subsequently, mixture No. 2 (**acetone + hydrogen + air**) was supplied, and after 5 min, the digital voltmeter reading was recorded again. The same procedure was performed for mixture No. 3 (**acetone + carbon monoxide + air**) and mixture No. 4 (**acetone + meth-**

ane + air), and the corresponding sensor responses were recorded.

For each standard gas mixture, the number of parallel measurements was $n = 5$. The mean results obtained for determining the selectivity of the SAS-Ac are presented in (Table 2).

According to the obtained data, the presence of carbon monoxide (up to **830 mg/m³**), hydrogen (up to **604 mg/m³**), and methane (up to **540 mg/m³**) in the analyzed acetone–air mixture does not affect the output signal of the acetone sensor.

Table 2. Results of the selectivity testing of the semiconductor acetone sensor SAS-Ac-3M ($n = 5$, $P = 0.95$)

Composition of the gas mixture	Acetone found, mg/m ³					
	SAS-Ac.No 1		SAS-Ac.No 2		SAS-Ac.No 3	
	$\bar{x} \pm \Delta x$	Sr10 ²	$\bar{x} \pm \Delta x$	Sr10 ²	$\bar{x} \pm \Delta x$	Sr10 ²
Acetone-563±(air, balance)	560±3	1,7	566±5	1,5	565±3	1,4
Acetone-563±H ₂ -604± (air, balance)	562±2	1,4	562±4	1,4	562±3	1,6
Acetone-563±CO-830± (air, balance)	559±3	1,6	565±3	1,5	562±4	1,6
Acetone-563±CH ₄ -540± (air, balance)	560±4	1,2	564±4	1,1	564±5	1,4

The data presented in (Table 2) demonstrate that, within the investigated concentration range, the developed sensor enables the selective determination of acetone. The measurement error of the SAS-Ac due to the presence of non-measured components does not exceed **1.5%**.

Thus, a sensor has been developed that ensures selective determination of acetone in multicomponent gas–air mixtures containing hydrogen, carbon monoxide, and methane (natural gas) simultaneously with acetone. Such mixtures include various natural and technical gas mixtures, as well as gaseous emissions from industrial enterprises.

In all cases, the relative standard deviation (**Sr**) due to the presence of non-measured components does not exceed **0.05**. Within the investigated flow-rate range of **5–50 L/h**, changes in the flow rate of the gas mixture do not significantly affect the output signal of the sensor.

The output signal of the sensors is also independent of their spatial orientation and inclination angle, which makes it possible to classify the developed sensors as **independent-type sensors in accordance with GOST 13320–82**.

Conclusion

Semiconductor methods with improved metrological characteristics have been developed, and on their basis, a semiconductor sensor has been developed for the determination of acetone in the atmospheric air of closed ecological systems and in human exhaled breath, with a measurement error not exceeding **1.0%**.

The oxidation mechanisms of combustible substances (**H₂**, **CO**, **CH₄**, and **acetone**) by atmospheric oxygen in the presence of metal oxides were investigated. Based on these studies, the optimal nanocomposite material for the gas-sensitive element of the semiconductor acetone sensor was determined to have the composition **SiO₂/(TiO₂ + 0.75CuO)**. The experimental results demonstrated the feasibility of developing highly selective semiconductor sensors based on nanocomposite materials exhibiting different levels of activity toward the individual components of gas mixtures. The main metrological characteristics of the selective semiconductor acetone sensor were evaluated.

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