E3S Web of Conferences **727**, 03005 (2026) https://doi.org/10.1051/e3sconf/202672703005
EEPES 2026

Measurement of hydrogen content in gas mixtures

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Abstract. Based on a comprehensive study of measuring the content for primary components in gases, a measuring transducer for determining hydrogen concentration in gas mixtures has been developed. This transducer is of significant importance for the oil and gas industry. Tests were conducted to determine the fundamental error of the hydrogen concentration transducer in air. Based on the experimental results, the actual values of the maximum absolute error for the output signal and the basic error were calculated, and static characteristics were obtained. The effects of temperature and pressure changes, as well as changes in hydrogen concentration, on the output signal and on the flow rate of the analyzed gas at the input of the primary measuring transducer were studied. The developed transducer was tested under constant experimental parameters, and it was found that the transducer output signal is not invariant to changes in the flow rate of the analyzed gas mixture. To determine the optimal load resistance, the influence of the external resistance of the electrical circuit on the transducer output signal was studied, and the corresponding characteristics were constructed.

1 Introduction

The problem of measuring hydrogen concentration in gas mixtures is of great importance to a number of petrochemical and refining industries [1-7]. Hydrogen is widely used in various technological processes - such as hydrotreating, hydrocracking, catalytic reforming, and ammonia synthesis - where its content directly affects reaction efficiency, catalyst durability, and final product quality. Insufficiently accurate or delayed determination of hydrogen concentration can lead to a decrease in the yield of target products, accelerated deactivation of catalysts, and the occurrence of emergencies, including explosive conditions, since hydrogen has an extremely wide explosive range (4÷75% by volume in air) and low ignition energy. Furthermore, in modern production environments, increasing attention is being paid to energy efficiency and environmental safety. Precise control of hydrogen content allows for optimized reagent consumption, minimized emissions, and reduced overall operating costs. However, measuring hydrogen in complex gas mixtures containing hydrocarbon vapors, hydrogen sulfide, ammonia, and other components presents a significant technical challenge. Traditional methods, such as gas chromatography or thermal conductivity, are

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often unreliable in industrial settings due to drift, sensitivity to vibration, the need for frequent calibration, or the use of auxiliary reagents. In this context, the development and implementation of new types of primary converters and controllers - compact, reliable, resistant to aggressive environments, and requiring no liquid electrolytes, auxiliary gases, or oxidizers - is becoming particularly important [8]. Such devices can provide continuous, highly accurate, and safe hydrogen monitoring directly in the process gas flow, opening up new opportunities for automation and increased process resilience in the fuel, energy, and chemical industries. Several methods and instruments have been presented for measuring hydrogen and oxygen concentrations in various gas mixtures [9-13]. These methods are based on the principle of direct conversion of chemical energy into electrical energy in fuel elements [14]. Their peculiarity is the use of synthetic polymer membranes as electrolytes, which act as ionic conductors. However, these methods required an auxiliary gas, an oxidizer, to measure hydrogen concentration.

2 Materials and methods

Figure 1 shows a new type of primary measuring transducer developed by the authors.

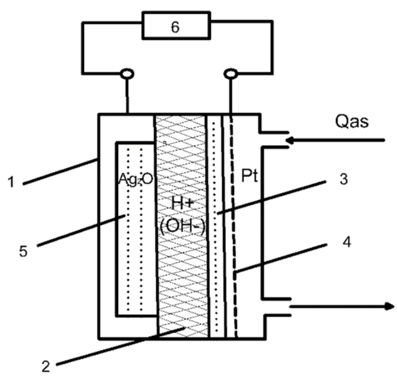

Fig. 1. Primary measuring transducer. 1 - transducer body, 2 - ion carrier, 3 - ion-exchange membrane, 4 - working electrode, 5 - auxiliary electrode, 6 - recorder.

The transducer body 1 contains working 4 and auxiliary 5 electrodes. The space between them is covered with a combined ion carrier 2 consisting of an OH-form ion-exchange membrane 3 and an OH-active ion-exchange resin. The output electrical signal from the transducer electrodes is recorded by 6 recorders. This electrochemical cell follows the following scheme:

Ag_2O - auxiliary electrode / carrier of H+ (OH-) ions / Pt - working electrode // gas mixture analyzed for hydrogen.

The converter is characterized by the absence of liquid electrolytes and auxiliary gases, small dimensions and mass, generative character of operation, resistance to vibration and shocks, and a number of other properties.

Experimental studies of the developed converters are carried out on a specialized test bench in accordance with the basic rules of international standards [15].

Tests to determine the basic error of primary measuring transducers of hydrogen concentration in air are carried out by alternately feeding the transducer input with gas mixtures of specified composition.

For testing, hydrogen-air gas mixtures were prepared and are summarized in Table 1.

Table 1. Hydrogen-air gas mixtures and their composition.

Mixture number	Mixture composition	Hydrogen content (%)
No. 1	Air	0.00
No. 2	Hydrogen-air	2.01
No. 3	Hydrogen-air	2.98
No. 4	Hydrogen-air	4.12
No. 5	Hydrogen-air	5.09
No. 6	Hydrogen-air	5.86
No. 7	Hydrogen-air	7.11

The error in the preparation and certification of gas mixtures does not exceed 1.5%.

For each gas mixture fed to the converter output, the value of the input signal on the scale of the automatic recording device is calculated 10 minutes after the beginning of feeding the next mixture. Then it is carried out.

Test conditions:

- Ambient temperature /gas mixture temperature/ - 20 ± 2ºC
- Atmospheric pressure - 101.03 kPa/758 mm. Hg.
- Relative air humidity - 75%
- Gas mixture flow rate - 3 ± 0.03 l/h
- External circuit resistance - 1 kOm

In the process of testing gas mixtures output value of the transducer is measured in the sequence specified in Table 2.

The results of tests to determine the basic error of the hydrogen concentration transducer in air are given in Table 3, taking into account the sequence of test steps given in Table 1.

To determine the basic error, the absolute errors of the transducer at each test point are initially taken into account according to the following equation:

$$\Delta U = U_u - U_2 , \quad (1)$$

where U_u is the measured value of the output signal in the controlled point, U_2 is the actual value of the output signal in the tested point.

The average algebraic number at each point is taken as the true (nominal) value of the converter output signal.

The value of the basic error is determined by the following formula:

$$\delta = \frac{\Delta U}{U_k - U_h} , \quad (2)$$

where U_k and U_h are the values of output signals corresponding to the end (0% H_2) and the beginning (6% H_2) of the measuring range of the hydrogen concentration transducer. The experimentally determined values of U_k and U_h are organised as follows:

U_k = 40.29 mB; U_h = 0.18 mB.

Calculation results for the actual values of the maximum absolute error of the output signal and the values of the basic error are given in Table 4.

Table 2. Sequence of test stages.

Stage No.	Sequential stages of hydrogenconcentration change in hydrogen-air-gas mixture	Serial numbers of hydrogen-air-gas mixtures	Stage No.
1.	First straight line	1 – 2 – 4 – 5	1.
2.	First opposite integer	5 – 4 – 2 – 1	2.
3.	Second complete integer	1 – 2 – 4 – 5	3.
4.	Second opposite integer	5 – 4 – 2 – 1	4.
5.	First straight reduced	1 – 5	5.
6.	First counter reduced	5 – X	6.
7.	Second straight reduced	1 – 5	7.
Stage No.	**Sequential stages of hydrogenconcentration change in hydrogen-air-gas mixture**	**Serial numbers of hydrogen-air-gas mixtures**	**Stage No.**
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4.	Second opposite integer	5 – 4 – 2 – 1	4.
5.	First straight reduced	1 – 5	5.
6.	First counter reduced	5 – X	6.
7.	Second straight reduced	1 – 5	7.

Analysis of the results of these types of tests shows that the maximum basic error in mixture No. 4 containing hydrogen (5.09 H_2) is 2.86%.

Table 3. Results of tests to determine the basic error of the hydrogen concentration transducer in air.

The number of hydrogen-air mixtures and the value of H2 concentration in them	Considering the sequence of steps value of H2 concentration in air						
	Step No. 1	Step No. 2	Step No. 3	Step No. 4	Step No. 5	Step No. 6	Step No. 7
Mixture No. 1 0% H_2	0.00	0.22	0.22	0.28	0.28	0.21	0.21
Mixture No. 2 2.01% H_2	17.85	18.54	18.72	19.45	-	-	-
Mixture No. 4 4.12% H_2	30.61	31.42	31.24	32.41	-	-	-
Mixture No. 5 5.09% H_2	35.81	35.81	37.72	37.72	36.43	36.43	36.32

Table 4. Calculation results for the actual values of the maximum absolute error of the output signal and the values of the basic error.

Row No.	Concentration of H2 in mixture, %	The actual value of the output signal, mV	Transducer error			
			Absolute maximum, %		Maximum error, %	
1.	0.00	0.18	0.16	0.00	0.39	0.00
2.	2.01	18.64	0.79	0.81	1.96	2.01
3.	4.12	31.42	0.81	0.99	2.02	2.47
4.	5.09	36.57	0.76	1.14	1.89	2.86

The effect of temperature change on the value of the output signal of the hydrogen concentration converter in air was tested according to the basic standard rules.

The range of temperature variation is within 0÷50^{0}C, so in the process of performing this stage of the test, changing the values of the output signal of the transducer, checking the ambient temperature is carried out by two values of hydrogen concentration with a change in temperature every 10^{0}C. The rest of the experimental conditions remain unchanged.

Table 4 shows the results of tests conducted under the following experimental conditions: pressure of the analysed gas mixture - 101.03 kPa/758 mm. Hg, mixture flow rate - 3 l/h, external circuit resistance - 1 kOm, composition of mixtures: in air mixture No. 2 - 2.01%, H_2 in air mixture No. 4 - 4.12%. Values of the converter output signals in Table 4 are taken as average values of the signals obtained at temperature decrease and increase:

$$\delta_t = \frac{\Delta U}{U_k - U_h} \cdot 100 . \quad (3)$$

Table 4 also shows the values of the converter error δ_t arising from the ambient temperature change by every 10^{0}C, calculated by formula (3). Here, U_k and U_h are the initial and final values of the converter output signals determining the conversion range.

ΔU_{t_0} is the change in the transducer output signal value at each point of the tested range of variation for every 10^{0}C change in ambient temperature:

$$\Delta U_t = \frac{U_t - U_{t_0}}{t - t_0} \cdot 10 . \quad (4)$$

Here, U_t and U_{t_0} are the values of output signals taken at temperatures *t* and to in 10^{0}C, taken when determining the basic error of the transducer. Based on the data in Table 5, the "Temperature-signal" characteristics is shown in Figure 2.

The effect of changing the pressure of the analyzed gas mixture on the value of the output signal of the hydrogen concentration transducer in air is tested on two test mixtures - mixture No. 2 with a hydrogen concentration equal to 2.01% H_2 and mixture No. 4 with a hydrogen concentration equal to 4.12% H_2. This is done using the following methodology.

The respective analyzed mixture is passed through the transducer, and its output signal is recorded as the pressure of the gas mixture changes in the transducer chamber.

During the test, the pressure of the analyzed gas mixture in the transmitter chamber is measured every 19.61 kPa/0.2 kg/sm^2 until the pressure is 199.08 kPa.

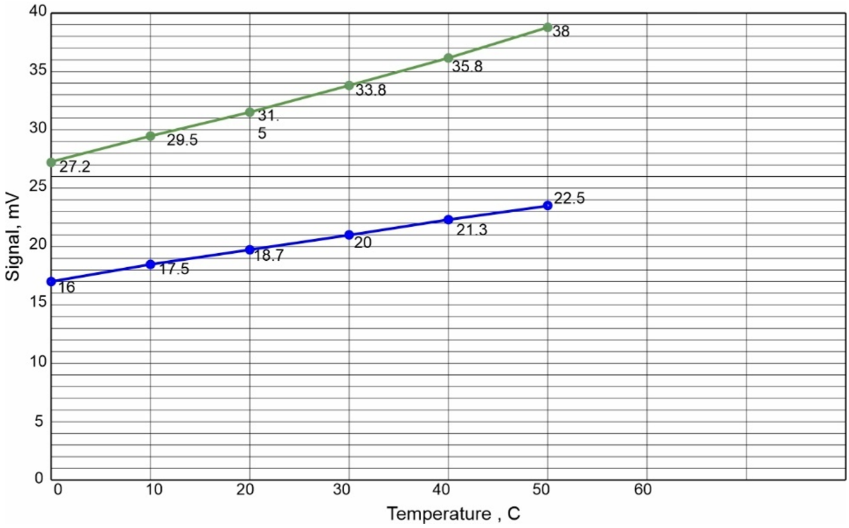

Fig. 2. Characteristics “temperature-signal”.

Four measurements are made at increasing and decreasing pressure values for each of the two analyzed mixtures. The results of the tests are shown in Table 5. Here, the values of the transducer output signal are taken as the average values of four signals obtained at increasing and decreasing pressures.

Table 5. Results of tests.

The value of temperature, ºC	The output signal of the transducer, mV		ΔU_t, mV		Additional error, δ_t %	
	Mixture No. 2	Mixture No. 4	Mixture No. 2	Mixture No. 4	Mixture No. 2	Mixture No. 4
0	16.04	27.17	1.33	2.18	3.31	5.43
10	17.38	29.37	1.32	2.17	3.29	5.41
20	18.70	31.54	0.0	0.00	0.00	0.00
30	19.99	33.72	1.29	2.18	3.21	5.43
40	21.27	35.90	1.28	2.18	3.19	5.43
50	22.54	38.06	1.28	2.17	3.19	5.41

The Table 6 also shows the values of additional error δp introduced by the transducer when the pressure of the analyzed gas in the working chamber changes every 19.61 kPa. This indicator is taken into account using the formula:

$$\delta p = (\Delta U_p)/(U_f - U_i) \cdot 100, \quad (5)$$

where U_f and U_i are the final (40.29 mV) and initial (0,18 mV) values of the output transducer signals, corresponding to the final (6% H_2) and initial (0.0% H_2) measurement transducer range, U_p is the value demonstrating the complete change in the output sensor signal at each tested measurement point for each change in pressure by 19.61 kPa within the specified limits.

This value is calculated using the following formula:

$$\Delta U_p = (U_p - U_{p_0})/(p - p_0) \cdot 19.61. \quad (6)$$

Here, U_p and U_{p_0} are the values of the output signals taken at pressures p and $p_0 = 101.03$ kPa, adopted when determining the basic converter error.

Table 6. Results of the tests.

The pressure value of the analyzed mixture, kPa	Output signal of the primary measuring converter, mV		ΔU_p, mV		Given additional error δp, %	
	Mixture 1	Mixture 2	Mixture 3	Mixture 4	Mixture 5	Mixture 6
101.03	18.58	31.64	0.0	0.0	0.0	0.0
120.64	22.11	37.68	3.53	6.04	8.80	15.06
140.25	25.83	44.92	3.62	6.64	9.02	16.55
159.86	29.35	50.94	3.59	6.43	8.95	16.28
179.47	33.55	57.20	3.74	6.39	9.32	15.93
199.08	36.79	62.01	3.64	6.07	9.07	15.13

Based on the data in Table 6, the “Pressure-signal” characteristics is constructed in Figure 3.

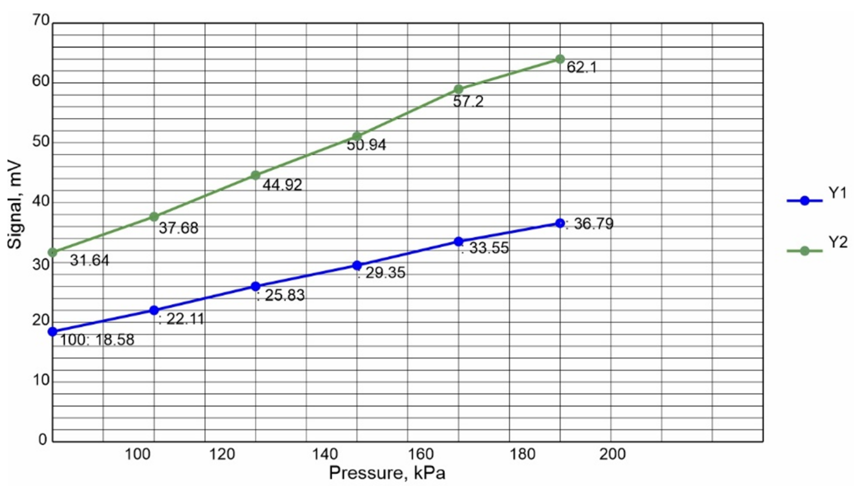

Fig. 3. Characteristics “pressure-signal”.

Analysis of the signal results shows that the output transducer signal largely depends on the pressure of the analyzed gas mixture in the working chamber of the transducer. This is due, on the one hand, to a change in the conditions of active substance transport to the reaction zone, and on the other hand, to a change in the properties of the ion carrier filling the space between the electrodes.

Taking this situation into account, new design variants of transducer layouts have been developed. Here, a pressure compensation unit for the gas mixture in the transducer working chamber is provided, as well as protection against pressure overloads. Subsequent tests of improved transducer samples have shown that the implementation of the above measures has made it possible to significantly reduce the influence of the analyzed gas mixture pressure on the transducer output signal.

The effect of changing the hydrogen concentration on the flow rate of the analyzed gas at the transducer input was tested by the conditions for determining the basic transducer error, and with the release of hydrogen-air gas mixture No. 2 with a hydrogen content of 2.01% H_2. Maintaining the specified flow rate was carried out using a gas flow regulator adjusted with an accuracy of 1%. Control of the flow rate for the specified gas mixture was carried out using a flow meter, i.e., a rheometer.

During the testing process, all the technological parameters of the experiment remain unchanged. The change required the flow rate for the analyzed gas in the range of 1÷10 *l/h* with a step of 1 *l/h*. Three measurements were taken at each specified value of the gas mixture flow rate.

Table 7 shows the stages of these tests. Test results analysis allows us to conclude that the transducer output signal is generally not invariant to changes in the flow rate of the analyzed gas mixture. However, as can be seen from Table 7, this effect manifests itself up to a flow rate of 8 l/h. From this point on, further changes in consumption have virtually no effect on the transducer output signal. Based on the data in Table 6, the "Gas flow-signal" characteristic is constructed in Figure 4.

Table 7. Stages of the carried-out tests.

The flow rate of the analyzed mixture, l/h	**The value of the output signal for the primary measuring transducer, mV**			**The average value of the output signal of the primary measuring transducer, mV**
	Test 1	**Test 2**	**Test 3**	
1	18.27	18.10	17.88	18.08
2	18.46	18.32	18.12	18.30
3	18.64	18.52	18.38	18.51
4	18.82	18.74	18.61	18.72
5	18.05	18.83	18.81	18.86
6	19.12	19.01	18.98	19.03
7	19.19	19.11	19.01	19.10
8	19.24	19.17	19.14	19.18
9	19.24	19.18	19.13	19.18
10	19.25	19.18	19.13	19.18

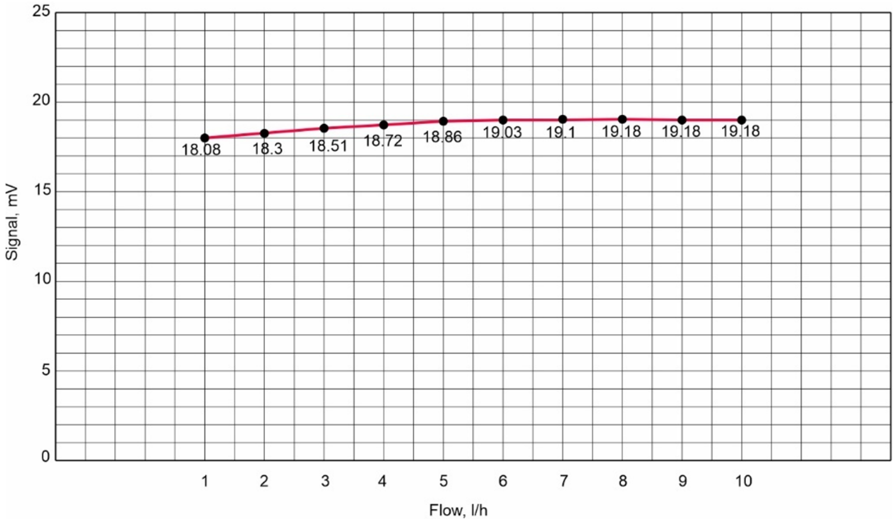

Fig. 4. Characteristic “Gas flow-signal”.

3 Results

The output transducer signal, which is a voltage drop across the load resistance, depends, all other things being equal, on the resistance value of the external electrical circuit and is determined by the hydrogen concentration in the analyzed gas mixture. It should be noted that increasing the value of the external circuit resistance (potentiometric mode) leads to a significant increase in the voltage conversion factor, but at the same time worsens the dynamic transducer properties, which leads to an increase in the full range of the indicator. In addition, the current mode of the transducer reduces the current transducer coefficient, improving its dynamic properties and ensuring compatibility with a low-resistance input secondary device. The above conditions require studies to determine the effect of the external electric circuit resistance on the output transducer signal (Table 8).

Table 8. Measurement results.

External circuit resistance, Ohm	The value of the output signal for the primary measuring transducer, mV			The average value of the output signal of the primary measuring transducer, mV
	Measurement No. 1	Measurement No. 2	Measurement No. 3	
100	2.36	2.18	1.99	2.18
500	9.63	10.18	9.45	9.75
1000	18.2	18.72	18.54	18.49
5000	58.18	59.90	60.01	59.36
10000	83.61	60.09	81.81	81.83

The objective here is to select the optimal value of the load resistance. During the tests, hydrogen-air gas mixture No. 2 with a hydrogen content of 2,01% H_2 is supplied to the transducer input, and its output signal is recorded at different values of the load resistance in the range from 100 Ohm to 10 kOhm. Three measurements were made at different resistance ratios set for each value of the external circuit resistance. The remaining test conditions correspond to the basic error definition.

Based on the data in Table 7, the "Load resistance-signal" characteristic is constructed in Figure 5.

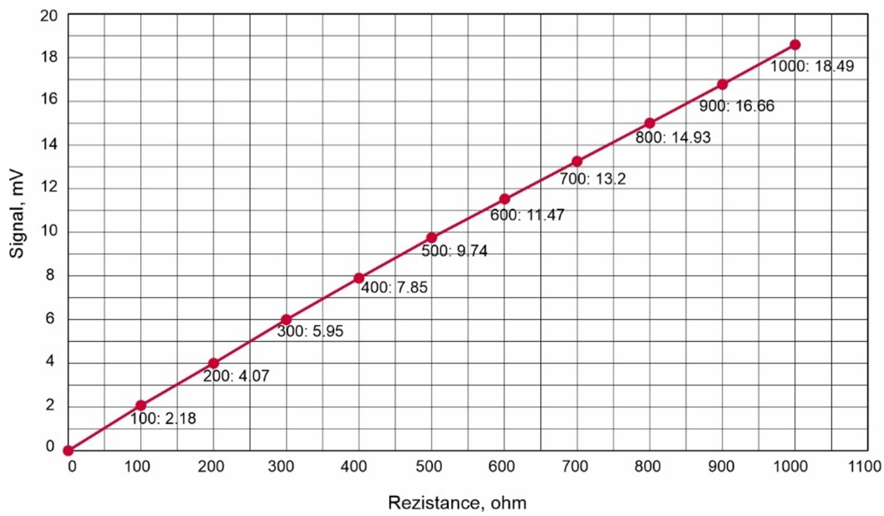

Fig. 5. Characteristic "Load resistance-signal".

4 Conclusion

The efficiency of an automated control system depends directly on the precise measurement of the composition and physicochemical parameters of the raw materials and the intermediate and final products by sensors of the operating technological installation. In the vast majority of technological processes, gases and vapors play the greatest role, and, given the specificity of their analytical methods, studying them and developing new gas analyzers are important and relevant. A new method for measuring hydrogen concentration in various gas mixtures has been proposed and studied. Its application has allowed us to examine the direct transfer of holes from chemical energy to electrochemical energy in the developed gas current generators, which occurs with the participation of quasi-solid polymer electrolytes.

As a result of the conducted tests and experimental studies, it was established that these transducers can be successfully used to solve large-scale problems related to the design of automated analytical monitoring systems and the creation of control systems for technological objects. The solution to this problem can be used to effectively solve a wide range of gas analysis problems. The results of the experimental studies fully confirm the transducer's performance and high metrological characteristics. These studies of advanced detectors make it possible to develop automatic gas analyzers based on them. Based on experimental results, the presented primary measuring transducer is capable of effectively solving the problem of gas analysis in the range of 0–6% hydrogen volume, with a measurement error of less than 3%. This range is determined by the linearity of the transducer's static characteristic within this interval. However, in some cases, it becomes necessary to measure hydrogen concentration over a wider range. Research has shown that reducing the hydrogen concentration in the reaction zone is necessary if the linear range of the primary transducer needs to be expanded. To achieve this goal, the analyzed mixture is not fed directly to the electrode-membrane system, but through a diffusion barrier, such as thin semi-permeable polymer films. This solution allows for a significant expansion of the measurement range, up to 100% of the hydrogen volume, and facilitates the implementation of these primary transducers in modern technological installations for the automatic analytical monitoring of hydrogen concentration in various gas mixtures.

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