RESEARCH AND CONTROL OF THE PURITY OF PRODUCTION HYDROGEN WITH A HIGH DEGREE OF PURIFICATION WHEN APPLYING THE ELECTROLYSIS METHOD OF PRODUCTION

На діючій електролізній установці на водневій станції отримання водню виконані дослідження чистоти продукційного водню високого ступеню очищення на вміст у ньому мікроконцентрації азоту на рівні 0,00005–0,0001 %. Для достовірності контролю чистоти технічного водню застосована цифрова фільтрація сигналу хроматографа, що заснована на байєсовському підході при мовах априорної невизначеності результатів досліджень. Визначена чистота отриманого продукційного водню на відповідність сертифікаційним показниками чистоти водню марки А

Ключові слова: електролізна установка, ступінь очищення, концентрація продукційного водню, домішка хроматографія, цифрова фільтрація

На действующей электролизной установке водородной станции получения водорода выполнены исследования чистоты продукционного водорода высокой степени очистки на содержание в нем микроконцентрации азота на уровне 0,00005–0,0001 %. Для достоверности контроля чистоты технического водорода применена цифровая фильтрация сигнала хроматографа, основанная на байесовском подходе при условии априорной неопределенности результатов исследований. Определенна чистота полученного продукционного водорода на соответствие сертификационным показателями чистоты водорода марки А

Ключевые слова: электролизная установка, степень очистки, концентрация продукционного водовода, примесь, хроматография, цифровая фильтрация

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1. Introduction

The last two decades have seen an ever-growing global interest in the development of new technologies for obtaining hydrogen. This is due, primarily, to the world energy crisis, in which hydrogen plays one of the leading roles as the fuel of the future [1]. Such a privileged position of hydrogen can be explained by the fact that hydrogen utilization is one of the possible solutions to the problem of scarcity of fossil fuels [2]. In addition, the use of hydrogen leads to zero environmental pollution by combustion products since the result of energy generation is such byproducts as heat and water only [3].

Hydrogen is widely used as a technological component in various industries: chemical and oil chemical (monomers production), energy generation (creation of autonomous stationary and mobile sources of energy to power fuel cells). Moreover, hydrogen is used in reducing metallurgy (anneal-

ing of special-purpose steels, alloying of powders, obtaining precision alloys), semiconductor, micro- and nanoelectronics, food, medical (manufacturing especially pure materials, substances and products) industries [4].

To implement a series of advanced promising techniques and technological processes, there is a need in obtaining high-purity hydrogen [5]. In this case, the need for ultrapure hydrogen of grade A for different purposes can vary widely [6, 7]. At the same time, ultrapure hydrogen has not been commercially available on the Ukrainian market for a long time [8].

The increasing demand for high-purity hydrogen with a purification degree of 0.000005–0.0002 % predetermines the minimum concentration of nitrogen in the commercial hydrogen of up to 0.0001 % [7].

In parallel with the need for modern technology of high purity hydrogen production, there is a need to devise and implement reliable methods to determine and control its purity. At present, there is no any single universal approach in world practice to defining and controlling the purity of production hydrogen [9].

It is an important scientific task to undertake research into determining the purity of hydrogen and the degree of its purification from admixtures during its commercial production.

2. Literature review and problem statement

There is no consensus among experts about the most cost-effective way to produce hydrogen [10]. Based on experimental data, authors of paper [11] conclude that the most affordable process, in terms of the cost of hydrogen production, is the conversion of methane. However, as noted in study [12], applying a given technology makes it possible to obtain hydrogen with a purity of 95–98 %, and, by employing additional energy-intensive purification technologies, it is possible to achieve a hydrogen purity of 99.99 %.

One of the most promising technologies for obtaining production hydrogen is the electrolysis technology [13]. Authors of paper [14] consider the electrolyzer to be one of the most potentially cost-effective techniques for hydrogen production. Electrolysis plants for the production of hydrogen are reliable, fully automated, environmentally safe, hydrogen generators, based on the technology of inorganic membrane (IMET) electrolysis of aqueous solutions of alkalis [15]. In the alkaline electrolysis, the reaction takes place in a solution consisting of water and a liquid electrolyte (30 % KOH) between two electrodes. At a sufficient voltage between two electrodes, H_2 molecules accumulate on the cathode while the anode, upon the passage of OH-ions through a 30 % KOH electrolyte solution, accumulates O_2 [16]. The resulting hydrogen undergoes further purification from water vapor and oxygen, the degree of which depends on the customer requirements. The purity of production hydrogen in this case reaches the values of 99.9998 % (pure 99.7 % oxygen is released into the air). The impurities in production hydrogen are only water vapor. If necessary, the purity of production hydrogen can be brought to 99.9998 % [17].

Thus, the most promising technology for the production of high purity technical hydrogen should be recognized to be a technology based on the application of electrolysis processes.

The main difficulty for manufacturing production hydrogen of high purity is the need to control purity of the product [18]. This task is resolved by developers of production technology in each case separately, taking into account technological features of the process and structural characteristics of equipment [19].

3. The aim and objectives of the study

The aim of present work is to undertake research into identification and control of hydrogen purity and the degree of its purification from impurities (nitrogen) in the microconcentrations of 0.000005–0.0002 %.

To accomplish the aim, the following tasks have been set:
– to develop a method to determine and control the purity of hydrogen and the degree of its purification from impurities (nitrogen) in the microconcentrations of 0.000005 0.0002 % using modern measuring devices;
– to carry out experimental studies in order to determine the purity of production hydrogen at the acting electrolysis installation manufactured by firm Hydrogen Technologies (Norway);
– to apply digital filtering of the chromatograph signal in order to improve the reliability of control over purity of technical hydrogen;
– to devise recommendations for correcting or adjusting the electrolysis installation in case the purity of production hydrogen does not match characteristics specified in technical documentation for the equipment.

4. Description of technology for obtaining production hydrogen with a high degree of purification

One of the global leaders in the manufacture of electrolysis installations for obtaining hydrogen, Hydrogen Technologies (Norway), built and put into operation the hydrogen station at the pipe plant Centravis Production Ukraine in the city of Nikopol (Ukraine). The station supplies production technical hydrogen for the process of heat treatment of pipes in a protective atmosphere, which in this case is the ultrapure hydrogen.

Fig. 1 shows technological circuit for obtaining production hydrogen with a high degree of purification.
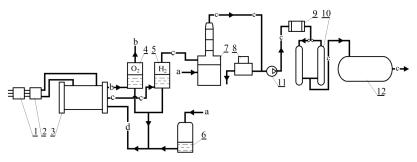

Fig. 1. Technological circuit for obtaining production hydrogen: 1 — transducer; 2 — rectifier; 3 — electrolyzer; 4, 5 — gas-alkali separator; 6 — alkaline container; 7 — scrubber; 8 — gasholder; 9 — purification; 10 — drying; 11 — multistage compressor; 12 — receiver; a — water; b — oxygen; c — hydrogen; d — electrolyte

Hydrogen forms as a result of electrolysis when electric current passes through the electrolyte (25 % aqueous KOH solution). In this case, water molecules are split at a ratio of 2:1 with oxygen emitted into the atmosphere while hydrogen, after purification, is fed to the technological circuit [20].

Table 1 gives technical characteristics of the atmospheric electrolyser produced by Hydrogen Technologies (Norway).

Table 1

Technical characteristics of atmospheric electrolyser

Parameter name	Value
Output of hydrogen, H_2, nm³/h	10−485
Maximal output of H_2 per cell, nm³/h	2.11
Electrical energy consumption at a load of 4000 ADC, kW·h/ nm³	4.1±0.1
Electrical energy consumption at a load of 5150 A DC, kW·h/ nm³	4.3±0.1
H_2 purity, %	99.9±0.1
O_2 purity, %	99.9±0.1
H_2 purity after purification, %	99.9998
H_2 pressure at the outlet from electrolyser, kPa	43.2
Maximal H_2 pressure at the outlet from electrolyser, kPa	1.46±4.91
Working parameters	
Working temperature, ºC	80
Output range	Automatic, 20−100 % of maximal output
Electrolyte	25% aqueous KOH solution
Water feed, l/nm³ H_2	0.9

In the process of operation of the electrolyser, the firm, which is the developer and manufacturer of the installation, faced a task to study the purity of produced hydrogen. The aim of the study was to determine characteristics of the product and to verify if parameters of the obtained hydrogen matched the specifications. It was also necessary to establish influence of technological parameters of the production process on the degree of purity of production hydrogen.

5. Examination of purity of production hydrogen with a high degree of purification

In the absence of any data on the level of pollution of production hydrogen with nitrogen at the outlet of electrolyser, examination of the purity of production hydrogen with a high degree of purification involved 2 stages:

– research into the presence of nitrogen in the samples of production hydrogen in the microconcentrations of [[N$_2$] 0.001−0.01 % (rough estimate);

– research into the presence of nitrogen in the samples of production hydrogen in the microconcentrations of [[N$_2$] 0.001−0.01 % (fine assessment).

The first stage provides for an assessment of the range of variability of nitrogen microconcentration in production hydrogen, predetermined by the technology of obtaining and the degree of hydrogen purification in the electrolyser itself. The second stage implies correction of the accuracy

in determining the concentration of [N$_2$], obtained using a thermal conductivity detector (TCD).

Nitrogen microconcentration in the selected samples was determined at the chromatographer Agilent Technologies G890 N with a TCD detector. The measurements of nitrogen microconcentration in production hydrogen (fine assessment) were refined using a more sensitive helio-ionization detector (HID).

The content of examined components in technical hydrogen was estimated applying a method of absolute calibration. When quantifying the content of impurities in hydrogen, we measured chromatogram peak areas. In line with a given method, we established a direct proportional dependence between the area of impurity peak and content in the chromatographed sample: $S=km$, where S is the area of peak on the chromatogram, m is the mass of a given component in the sample, k is proportionality coefficient. The peaks areas were measured on the chromatogram using the integrator. The chromatogram peak area measurement error did not exceed 1 %.

Based on the data obtained, we constructed calibration diagrams in coordinates S_i−C_i (area − mass content of substance in a sample (%)) for a series of model solutions with a precisely known concentration of C_i impurities in technical hydrogen. Next, strictly under the same conditions, we chromatographed hydrogen samples with an unknown concentration of C_{H2} and measured the area of peaks of oxygen and nitrogen. The area of peaks of oxygen in the examined samples were calculated in order to verify a version of possible air penetration into the system while taking produced hydrogen into cylinders. Based on the calibration diagram, we determined the concentration of C_{N2} of the examined substance in the analyzed solution. The concentration of C_{H2} was determined by estimation.

To implement the specified stages, applying a gravimetric method using the scale made by Mettien-Toledo and Sartorius, we prepared reference model mixtures of hydrogen. Molecular part of nitrogen in hydrogen was 0.00464 % and 0.00234 %. respectively. Such concentrations corresponded to the first and second points for constructing the calibration curve, characterizing the microconcentration of nitrogen in the examined hydrogen. The third point for constructing the calibration curve, characterizing the microconcentration of nitrogen in the examined hydrogen, was high-purity hydrogen (1.0 cm³/m³) manufactured by Linde Gas (Germany). Indicators of the certified reference hydrogen corresponded to the purity of 99.9999 % with a molar fraction of nitrogen C_{N2} in it of 0.00010 %.

Prepared mixtures were exposed to homogenization for 72 hours, after which they were examined at the gas chromatographer Agilent Technologies G890 N equipped with a helio-ionization detector (HID).

The calibration curve for the distribution of nitrogen microconcentrations in hydrogen, obtained using the TCD detector, was derived in the same way. We used, as benchmark reference mixtures, hydrogen mixtures with molecular fractions of nitrogen in hydrogen and fractions of 0.525 % and 0.046 % respectively. The third point for constructing the calibration curve was high-purity hydrogen manufactured by Linde Gas (Germany) with a molar fraction of nitrogen C_{N2} in it of 0.00010 %.

Fig 2, 3 show calibration curves for the distribution of nitrogen microconcentrations in hydrogen, acquired from a TCD detector and a HID detector, respectively. The curve

shown in Fig. 2 is approximated by equation of the form $y=2312.9x-0.0016$ with regression coefficient $R^2=1$. The curve shown in Fig. 3 is approximated by equation of the form $y=283.57x-27.518$ with coefficient $R^2=0.9999$ [21].
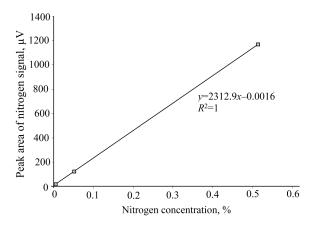

Fig. 2. Calibration curve for the distribution of nitrogen microconcentrations in hydrogen acquired from a TCD detector
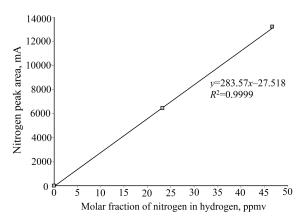

Fig. 3. Calibration curve for the distribution of nitrogen microconcentrations in hydrogen acquired from a HID detector

Regression coefficients of the calibration curve (Fig. 2) were determined using a regression analysis tool embedded in the software MS Excel, Analysis Tool add-in (Fig. 4).

Results of the computation of regression coefficient and the approximation of calibration curves are shown in Fig. 5.
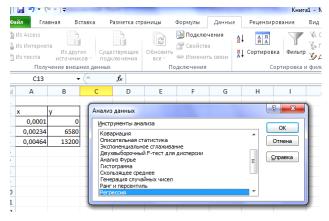

Fig. 4. Regression analysis tool window

Regression coefficients of the calibration curve (Fig. 3) were determined in a similar way.

For the purpose of study, production hydrogen was taken as samples to specially prepared (pressurized in advance) cylinders with a volume of 10 l in the representative points of the industrial installation for manufacturing production hydrogen. The cylinders were normalized. Before taking the samples, the lines that connect cylinders to the sampling points, as well as the cylinders themselves, were blown for 30 minutes by production hydrogen, to prevent any contaminants penetrating the examined samples.

To perform the research at the chromatograph Agilent Technologies G890 N, we took out 5 injections from each of the normalized cylinder in order to analyze them using the chromatograph. Prior to the analysis, the chromatograph and the lines that connect the cylinder to the chromatograph were "washed" for 10 minutes with production hydrogen from the examined cylinder. Upon completion of the "washing" process, we sequentially introduced injections to the chromatograph in order to determine the microconcentrations of nitrogen and hydrogen. Exposure time corresponded to the maximum nitrogen concentration, registered at the chromatograph display: it was ≈4 minutes.

The area of microconcentrations peak was calculated in μV. The analysis was performed until the peak indicator reached stability at the display. We simultaneously controlled the peak of oxygen concentration in the examined samples upon expiring the exposure time of 2 minutes.

ВЫВОД ИТОГОВ

Регрессионная статистика	
Множественный R	0,999
R-квадрат	0,999999989
Нормированный R-квадрат	0,999930841
Стандартная ошибка	1,88784E-05
Наблюдения	3

Дисперсионный анализ

	df	SS	MS	F	Значимость F
Регрессия	1	1,0306E-05	1,0306E-05	28917,6747	0,003743634
Остаток	1	3,56393E-10	3,56393E-10		
Итого	2	1,03064E-05			

	Коэффициент	Стандартная ошибка	t-статистика	P-Значение	Нижние 95%	Верхние 95%	Нижние 95,0%	Верхние 95,0%
Y-пересечение	-0,0016	1,7223E-05	5,357332264	0,117479534	-0,00012657	0,000311109	-0,00012657	0,000311109
Переменная X 1	2312,9	2,02258E-09	170,0519765	0,003743634	3,18244E-07	3,69642E-07	3,18244E-07	3,69642E-07

Fig. 5. Results of computation of regression statistics

A signal from the chromatograph was processed using a developed algorithm of digital filtering. The proposed noise filtering algorithm enables the minimum possible confidence interval for each point of a chromatogram. Structure of the model was designed to process noisy results of measuring vibration-frequency converters [21]. The model is based on the Bayesian approach that employs *a priori* information about model's parameters. This information is represented in the form of *a priori* probability, or a probability density function. Next, the initial probabilities are "reviewed", using sample data, which are mapped in the form of *a posteriori* distribution of estimates of parameters or variables. The case of practical implementation of the algorithm for filtering the results of pilot tests of a vibration-frequency sensor in the programming environment Python (Python Software Foundation License) is given in paper [22]. The model was further developed and adapted to work with arrays of data acquired from chromatogram processing.

The obtained model of the developed algorithm for digital filtering of a chromatograph implies the following: a model for data gathering is created, which depends on the measured parameter. In this case, the purpose is data interpolation using a straight line in the form $y=ax+b$ (we assume that all data have a linear dependence with the Gaussian noise, imposed on it, with a known variance). Then a and b are the parameters whose probable values need to be determined while a likelihood function is the Gaussian function with the mean defined by the equation of a straight line and a given variance. *A priori* probability includes information known prior to performing the analysis. For example, it is known that the straight line should have a positive slope, or that the value at the point of intersection with the y axis must be positive, all this is accounted for in the proposed model. When assigning *a priori* distribution, we set the first argument, as well as the boundaries of distribution (the second and third arguments).

Processed results of research are given in Table 2.

Analysis of the data given in Table 2 reveals that the averaged value of area of [N$_2$] peaks in the examined samples, when using a thermal conductivity detector (TCD), is 286.04 µV. Based on the calibration dependence of area of the nitrogen microconcentration peak in hydrogen, we determined nitrogen concentration in production hydrogen.

At the same time, the averaged value of oxygen peak area in the examined samples was 4.54 µV.

The microconcentration of nitrogen in the examined hydrogen was on average 0.1 %, which indicates the purity of production hydrogen at a level of 99.9±0.1 %.

Table 2

Results of research into nitrogen and oxygen microconcentrations in the samples of production hydrogen

No. of cylinder	No. of sample	Area of peak N$_2$ in injection, µV	Area of peak O$_2$ in injection, µV
1	1	308.1	4.80
	2	309.0	4.80
	3	309.8	4.79
	4	309.8	4.80
	5	309.8	4.80
2	1	295.3	4.49
	2	280.0	4.50
	3	279.2	4.50
	4	279.0	4.49
	5	279.0	4.50
3	1	285.3	4.50
	2	284.9	4.55
	3	280.3	4.49
	4	280.1	4.50
	5	280.1	4.50
4	1	279.7	4.45
	2	278.9	4.44
	3	278.6	4.40
	4	278.0	4.41
	5	278.1	4.41
5	1	278.1	4.46
	2	277.9	4.46
	3	278.0	4.45
	4	277.0	4.45
	5	277.0	4.45

6. Discussion of results of examining the purity of production hydrogen with a high degree of purification

Studying the purity of production hydrogen with a high degree of purification for its content of nitrogen microconcentration is required to address the challenges of shortage of fuel, extending the element base of nanoelectronics. The lack of proven procedure for investigating the purity of production hydrogen with a high degree of purification significantly inhibits the development of Ukrainian production of high purity hydrogen.

The proposed method for determining and controlling the purity of production hydrogen includes two stages of research at a chromatograph: rough estimate of the presence of nitrogen in the samples of production hydrogen in the microconcentrations of [N$_2$] 0.001–0.01 % using a TCD detector, and fine assessment of the presence of nitrogen in the microconcentrations of [N$_2$] 0.00005–0.0001 % applying a HID detector.

The reliability of control is ensured by a special procedure for taking samples (preparation of cylinders, selection of representative sampling points, etc.). To improve the reliability of control, we a used digital filtering algorithm when processing results of chromatograph measurements.

Ratio of the averaged area of oxygen and nitrogen peaks in the examined samples was 4.54/286.04=0.016; the percentage of oxygen and nitrogen concentrations in ambient air was 21/79=0.27 [19]. This indicates that there is no air

in the samples of the production hydrogen. That is why at the second stage of measurements we determined nitrogen concentration in hydrogen.

The research data obtained allow us to argue that the purity of production hydrogen is within 99.9±0.1 %. This does not match certification indicators for the purity of production hydrogen claimed by the manufacturer to equal 99.9998 %.

The high level of nitrogen microconcentration in hydrogen is caused by worn piston rings of compressor pistons. Leakage in piston rings causes the penetration of nitrogen in microconcentrations into production hydrogen.

In accordance with the technological regulations, production hydrogen, prior to entering the receiver, is compressed in a multi-stage piston compressor. The number of stages in the compressor is predetermined by the degree of compression of production hydrogen. The compressor is cooled when gas passes from one stage to another. Cooling of piston compressor is performed by technical nitrogen at a pressure of 10 MPa. The high pressure of nitrogen and the lack of air-tightness in the compressor's cooling system due to the wear during operation of piston rings in the compressor's stages leads to an increase in the microconcentration of nitrogen in production hydrogen. This affects the purity of hydrogen.

To confirm the hypothesis put forward, we replaced piston rings in the stages of the compressor. After replacing the piston rings, we undertook new research into the purity of production hydrogen applying the technique described above. As a result of research, the purity of production hydrogen at a level of 99.99±0.01 % was registered, which corresponds to the hydrogen of grade A [23].

The developed method of control over purity of production hydrogen has now been introduced to the enterprise's technological process regulations. A given method could be employed to determine the purity of production hydrogen obtained using the electrolysis technology, as well as for other manufacturing technologies.

7. Conclusions

1. We conducted experimental studies aimed at determining the purity of production hydrogen at the electrolysis installation made by Hydrogen Technologies (Norway) at the pipe plant Centravis Production Ukraine in the city of Nikopol. Determination of hydrogen purity and the degree of its purification from impurities (nitrogen) in microconcentrations was carried out in two stages:

– research into the presence of nitrogen in the samples of production hydrogen in the microconcentrations of $[[N_2]$ 0.001−0.01 % (rough estimate);

– research into the presence of nitrogen in the samples of production hydrogen in the microconcentrations of $[[N_2]$ 0.001−0.01 % (fine assessment).

2. We determined that the purity of production hydrogen was 99.9±0.1 %. A given value for purity does not match certification indicators for purity of production hydrogen claimed by the manufacturer to equal 99.9999 %.

3. We analyzed the reasons for the mismatch between the registered purity of production hydrogen and claimed characteristics. Analysis of the results of research revealed that the cause of high nitrogen concentration in hydrogen is the worn piston rings in the stage of compressor pistons. Leakage causes the penetration of nitrogen in microconcentrations into production hydrogen.

4. Piston rings in the compressor's stage were replaced. We conducted repeated studies into purity of production hydrogen. It was found that the purity of production hydrogen in the repeated tests amounted to 99.99±0.01 %, which corresponds to the hydrogen of grade A and meets technological requirements of the customer.

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