

*Досліджуються процеси термоокислительного розкладання відходів на прикладі синтетичного волокна нітрону. Досліджено можливість формування нановолокон нітрону. РЕМ-зображення демонструє упаковані нанонитки, діаметром 50...150 нм, при яких імовірно стає прояв квантоворозмірних ефектів. Експериментально встановлено газоподібні речовини і їх концентрація залежно від температури протікання процесу*

*Ключові слова: відпрацьовані наноматеріали, термоокислювальне розкладання відходів полімера, атмосферне повітря, ідентифікація токсичних речовин*

*Исследуются процессы термоокислительного разложения отходов на примере синтетического волокна нитрона. Исследована возможность формирования нановолокон нитрона. РЭМ-изображение демонстрирует упакованные нанонитки диаметром 50...150 нм, при которых вероятно становится проявление квантоворазмерных эффектов. Экспериментально установлены газообразные вещества и их концентрация в зависимости от температуры протекания процесса*

*Ключевые слова: отработанные наноматериалы, термоокислительное разложение полимера, атмосферный воздух, идентификация токсичных веществ*

# RESEARCH OF THE INFLUENCE OF DECOMPOSITION OF WASTES OF POLYMERS WITH NANO INCLUSIONS ON THE ATMOSPHERE

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## 1. Introduction

The presence of harmful and dangerous gases, as well as suspended particles and aerosols in the atmosphere, poses a serious threat to biological diversity and human health. Constant increase in their concentration leads to a decrease in the level of environmental safety.

A state of health and life expectancy of the population depends on the level of environmental safety in general and the quality of atmospheric air in particular.

Increased demands on materials and products made of them explain creation of new technologies and industries. At the same time, harmful emissions into the atmosphere [1] and formation of waste with the subsequent accumulation of heavy metals in the ground [2] accompany each technological process. Despite the availability of modern technologies for solid waste management [3–8], which makes it possible

to obtain energy raw materials [9, 10], the most frequent treatment is burial. This fact contributes to:

- thermo-oxidative decomposition of polymeric synthetic waste materials with a release of decomposition products into the atmosphere;
- migration of waste nanoparticles containing waste nanomaterials to the atmosphere.

A problem of waste is relevant in many countries [3–8]. Places of their accumulation pertain to anthropogenic objects. Such places are hidden sources of ecological danger formation. They require special control. It is impossible to identify them with the naked eye as mentioned processes occur at some depth of a waste massif. Processes develop in limited time intervals in territories with finite sizes. Given this, there is a high intensity of formation of dangerous molecular compounds in the atmosphere [11] and occurrence of unintentional fires.

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## 2. Literature review and problem statement

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As a rule, morphological and component composition of waste is very different depending on a region, a settlement, and a season. Significant parts of waste are:

- polymeric materials – elements of product construction, plastic packaging, clothing, etc.;
- nanotechnology products – most consumer products like cosmetics, antibacterial textiles, lithium-ion batteries, glass coatings, transistors, photodiodes, etc.

Analysis of literature sources showed that polymeric synthetic materials make up 40...60 % of the total mass of waste [6, 12].

Presumably 10 % are engineering nanomaterials (ENM) [13]. The quantity of nanomaterials is constantly growing in household goods [14] and they also get into waste after the end of their service life. People transport approximately 20,000 tons/year of nanocomposites to landfills as wastes according to some estimates [15]. Nanomaterials traditionally include objects, one of characteristic sizes of which lies in the range from 1 to 100 nm. That is, almost every synthetic material can contain particles of nanometer size.

Processes of thermo-oxidative decomposition of synthetic materials occur under conditions with variable climatic parameters. A release of toxic substances accompanies the processes. At the same time, under conditions of considerable height of waste mass, access to oxygen is very limited or absent, which leads to incomplete oxidation of gases. A study [16] says that products of incomplete combustion release themselves in waste storage sites. They include polyaromatic hydrocarbons, methanol, formaldehyde-based aldehydes, various carboxylic acids, amino acids, etc. However, we do not know under which conditions their release begins.

A paper [17] states that thermal decomposition of polymers produces methane, hydrogen, ethane, ethylene, formaldehyde, carbon monoxide and others. Smoke and toxic gases such as carbon monoxide, hydrogen cyanide, nitrogen oxide, phosphines, phosgene, and hydrogen chloride release themselves in the combustion process. At the same time, there is no indication of temperature intervals for the release of mentioned substances and their intensity. Another work [18] studies thermal decomposition of synthetic materials to obtain a modified fiber to reduce the combustibility of a material.

In turn, toxicity of nanomaterials is connected with admixtures present in them considerably [19]. Currently we understand consequences for the safety of the environment poorly due to special chemical and physical characteristics of nanomaterials [20].

A danger of nanomaterials lies also in the fact that nanoparticles can have a significant number of broken bonds, which can attach free radicals [21].

Synthetic materials with nanoinclusions can contain carbon fibers [22], carbon nanotubes [23], silica nanoparticles [24] and metal oxides [25]. Authors of papers [15, 26] investigated the ecological hazard of such materials after the end of their service life. They developed an integrated system for thermal decomposition of carbon nanotubes under controlled conditions at high temperatures – 800 °C and above. However, according to long-term observations of landfill sites, researchers established that the temperature in the thickness of a landfill remains at 20...80 °C according to the source [27] and 20...65 °C according to a work [28].

A moisture content of waste depends on a season mostly and fluctuates between 40...80 %.

Some authors believe that we must have identification methods in ecological and biological systems to ensure safety of nanomaterials [29]. The mentioned methods should be able to identify nanomaterials specifically. This is justified by the fact that molecular and bulk shapes of materials containing the same elemental profile will have different physical and chemical properties [30, 31].

Authors of paper [32] show that we cannot apply standard toxicological methods to the determination of a danger of nanomaterials. Because their properties depend not on concentration only, but also on specific quantum-dimensional properties [33]. Thus, we can state that many scientists point to a potential danger of polymer materials and nanotechnology products for the environment and human health. In addition, modern science focuses on such issues as: a life cycle of goods made of mentioned materials [34, 35], methods for evaluation of a quality of nanostructures [36], disposal of polymers [3, 7] and nanomaterials [37].

An increase in temperature in a body of waste deposit polygons from 20 °C and above activates activity of microorganisms. When the temperature reaches 65...70 °C, microorganisms die, but oxidation processes have already begun and proceed with a release of heat. In the case when heat dissipation exceeds heat extraction, there is a self-heating of substance, an intensification of the oxidation process, its self-acceleration and further self-heating of a material and self-ignition. As a result of such processes, the temperature in a body of a polygon can rise significantly above 80 °C. This leads to hidden processes of combustion, thermal decomposition of synthetic materials, including materials with nanoinclusions, and migration of environmentally hazardous substances into the natural environment. At the same time, conditions and duration of smoldering of various substances in the total mass of waste are not the same. There are no sufficient data on their investigations. Since we do not know time from the beginning of smoldering to the appearance of open fire, consequences for components of nature can be disastrous. And, consequently, it is necessary to search for methods of prevention of serious negative consequences at the earliest stages of the development of thermal decomposition of materials.

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## 3. The aim and objectives of the study

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The aim of present study is to analyze the possibility of determination of toxic substances, including nanoparticles, that enter the atmosphere during degradation, combustion (smoldering) of synthetic waste materials. This will provide an opportunity to justify a choice of a further way of the study on the environmental impact assessment and identification of a hidden danger in a body of a polygon.

We solved the following tasks to achieve the objective:

- the investigation on conditions for the migration of nanoparticles from waste that contains spent nanomaterials into the environment;
- experimental determination of concentrations of toxic substances that are released during thermal decomposition of synthetic polymeric materials;
- the substantiation of the possibility of high-accuracy identification of gaseous substances in the atmosphere in order to identify a hidden danger in a body of a polygon.

#### 4. Materials and methods of the study of gaseous products formed during polymer smoldering

We performed an experimental study on the example of one of widespread polymers – synthetic nitron fiber – to determine a composition of gaseous products formed during its decomposition.

We considered an imitation model, which is as close as possible to the actual conditions of a waste landfill to study conditions of migration of waste nanomaterials from waste to the environment. We heated the nitronic fibrous material in the temperature range of 150...300 °C in the presence of oxygen. We stretched the material under investigation until the appearance of fiber breaks. The time of the experiment varied from three to thirty minutes. We wound the fiber on a coil from both ends to provide tension and blew it simultaneously with a stream of air to remove liquid and gaseous phases. We dried the obtained skeins for three days. We studied the morphology of the resulting structures with a scanning electron microscope.

We took air-dry samples of nitron fiber, humidity 2.0...3.5 %, weighed 0.5 g (weighing error – 0.01 g) for the experimental determination of a concentration of toxic substances. We choose five samples kept in laboratory conditions at a temperature of 23 °C for 48 hours for the experiment. We heated the samples with an electric heater in a laboratory chamber. Asbestos cement plates of 20 mm thickness isolated the inner surface of the laboratory chamber. We observed thermal degradation of the nitron fiber in the temperature range of 100...600 °C in an inert medium and in the presence of air.

We carried out quantitative analysis of gas generation under constant experimental conditions and used a standard method of gas chromatography. We assumed that the intensity of a detector signal was directly proportional to a concentration of *i*-th component in a mobile phase, and the area of the corresponding peak in the chromatogram - to its amount. We sent a gas emanating from the laboratory chamber to Drechsel filled with 0.01 mol/l of sulfate and a solution of ammonium polysulphide. We took samples at each increase in the heating temperature by 50 °C to determine a composition of evolved gas by a gas meter. We transferred the components of the mixture of formed gases in a chromatographic column. We carried out a transfer of the sample from the dispenser to the chromatographic column by a gas carrier through an evaporator. We used argon as a carrier gas in the experiment, since it is the most common inert gas and it is effective in a process of investigation of decomposition products. We regulated the speed of gas carrier in the range of 3.0...3.6 ml/s. It was cleaned and dried in the gas preparation unit. We determined the amount of passing gas using a gas clock. We used aqueous solutions of 0.01 mol/l of sulfate and ammonium polysulphite to absorb ammonia and hydrogen cyanide respectively. We detected ammonia in samples on PhEC-M photocolormeter using Nessler reagent. We fixed gaseous products of thermal degradation of nitron fibers with a gas chromatograph LChM-8MD in laboratory conditions. To determine the effect of a heating rate of the polymer fiber on the practical output of cyanide hydrogen, the heating rate varied from 25 to 100 °C/min.

#### 5. Results of experimental studies on the thermal degradation of a synthetic polymer

A disorientation of macromolecules relatively to a fiber axis occurs when we heat fiber above 80 °C. At the same time, both intramolecular cyclization reactions and reactions of intermolecular crosslinking of polymer molecules take place. Fibers have different microstructures, which depend on conditions of formation and composition of the initial polymer. The microstructure in turn affects the interaction of a fiber with air oxygen upon heating. The more developed surface and the lower density of a fiber contribute to better penetration of air oxygen into the mass of material and makes possible the flow of chemical processes at lower temperatures. Fibers are thinning when we the material gets heated due to melting and stretching under an influence of the mass of waste from above. Thus, nitron fibers themselves become thin enough, and so they can be classified as nanofibers. Fig. 1 shows a photomicrograph of nitron fibers obtained as a result of the imitation experiment.

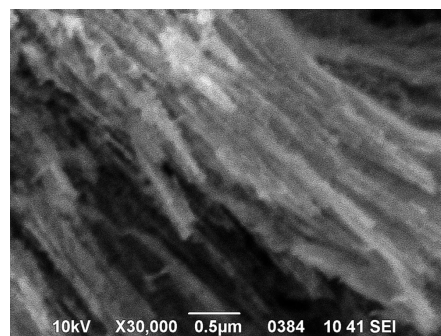


Fig. 1. REM-image of nitron nanofibers obtained by heating of the material to 200°C for 25 min

Fig. 1 shows a dense network, which consists of nanothreads with a diameter of 50 to 150 nm. Nanothreads are one-dimensional nanoobjects and can exhibit quantum-dimensional effects. The threads are tightly packed and have different lengths. This indicates instability and fragility of the material obtained. Fragility is a factor that is crucial for the spread of nanofibres in the environment as a whole, and in atmospheric air in particular. The smallest particles can easily be lifted by air masses and form a stable dispersed system. When going to nanoscales, a huge increase in the ratio of a number of particles and the total area of their surface to the mass occurs.

According to the results of experimental studies of the practical output of gaseous products during thermal degradation of synthetic fiber, we obtained data shown in Table 1.

We detected that gas formation accompanies the burning process (smoldering) of the initial synthetic fiber. The release of ammonia started at 100 °C, but at low concentrations (Fig. 2). The release of hydrogen cyanide in the form of bromo-cyan occurs in the presence of an oxidant at the same temperature, although its release begins at 250 °C only without oxygen.

A more intensive release of gaseous products accompanies processes in the oxidizing medium, while they slow down in the inert medium (Fig. 3). Factors that influence the decomposition process are, first of all, temperature and duration of heating.

Table 1

Practical output of gaseous products during thermal degradation of synthetic fiber (per 1 g of the test material)

T, °C	In the inert medium			In the presence of an oxidizing agent				
	H <sub>2</sub> , ml	NH <sub>3</sub> , mg	HCN, mg	H <sub>2</sub> , ml	NH <sub>3</sub> , mg	HCN, mg	CO, mg	CO <sub>2</sub> , mg
80	–	–	–	–	–	–	–	–
100	–	0.010	–	–	0.005	0.081	–	0.100
150	–	0.012	–	–	0.006	0.377	–	0.368
200	–	0.026	–	–	0.017	0.402	–	1.000
250	–	0.059	1.000	–	0.031	2.435	0.221	2.410
300	0.001	0.093	1.203	0.001	0.045	3.841	0.545	3.109
350	0.030	1.001	3.225	0.001	0.510	6.791	1.224	4.706
400	2.200	0.836	3.817	0.020	0.378	7.500	2.101	6.500
500	8.354	0.050	4.154	0.590	0.001	8.434	3.081	9.872
600	13.831	0.021	7.120	4.520	0.000	14.020	5.090	15.410

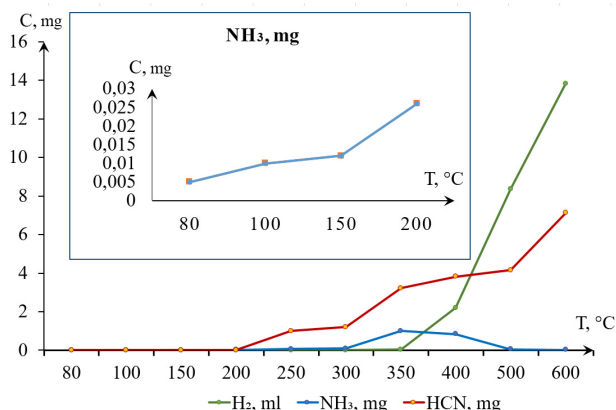


Fig. 2. Dependence of a concentration of toxic substance on its heating temperature without an oxidant

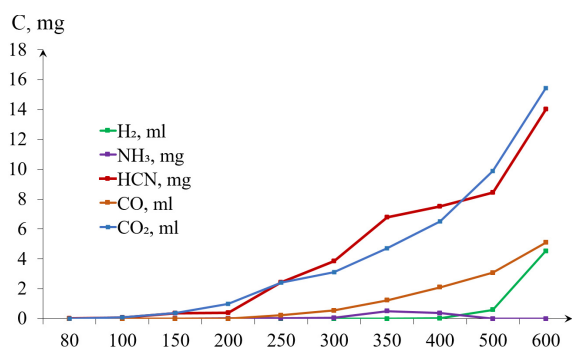


Fig. 3. Dependence of a concentration of toxic substance on its heating temperature in the presence of an oxidant

The process temperature must be the higher the shorter is heating time to achieve a certain degree of decomposition. Long-term heating contributes to the occurrence of side reactions. The main indicators of hidden combustion processes (smoldering) of materials can be such gaseous products of thermal degradation of synthetic fiber as hydrogen, hydrocyanic acid, ammonia, oxide and carbon dioxide.

Identification of the mentioned gaseous substances in the atmospheric air above a landfill may indicate the imminent realization of an emergency. In this connection, the question arises of a method for identification of the mentioned

substances in the atmosphere above a polygon under the following conditions:

- 1) very low concentrations of gaseous indicator substances in the early stages of combustion (smoldering);
- 2) a significant size of a polygon area and a danger of failure of a waste massif.

### 6. Discussion of the results and prospects of the experimental study conducted

The study of development of processes of thermo-oxidative decomposition of a polymeric fiber showed the following. We observed thinning of fibers of the synthetic material under the influence of the mass of waste and simultaneous heating of the material. This gave grounds to classify it as a nanofiber from a certain moment. This fact can influence a further rate of development of the thermo-oxidative decomposition of the fiber significantly. Since a high specific surface area of nanomaterials increases their adsorption capacity, chemical reactivity and catalytic properties [38, 39]. In addition, nitron can contain nanoparticles of various compounds, in particular silver, iron, arsenic, cobalt [40, 41]. The presence of nanostructured inclusions in a polymer leads to the appearance of quantum-dimensional effects, which change properties of matter fundamentally [42]. Thus, another danger lies in the increase in production of free radicals and active forms of oxygen in the mass of nitronic material with the nanoparticles. Active forms of oxygen stimulate oxidation processes, which, in turn, leads to self-heating and self-ignition of polymer waste.

Experimental studies of gas release during fiber smoldering let us suggest that the main substances-indicators of hidden combustion processes (smoldering) of polymeric materials in a body of a landfill are ammonia and hydrogen cyanide. However, the studies tested the most widely known materials. Therefore, in the long term, we need a more detailed analysis carried out by modeling processes of thermo-oxidative decomposition for a mixture of different materials, which most fully corresponds to the current composition of waste. At the same time, serious attention should be paid to the influence of nanoparticles on thermo-oxidation processes of waste materials and a composition of released substances. We need to have a high-precision gas analysis method to prevent the development of an emergency as a release of gaseous toxic substances occurs at 100 °C already, but at low concentrations.

Such a gas analysis can be performed with a use of a laser complex. The frequency-tunable laser complex provides the ability to determine resonance frequency of light absorption sequentially in the infrared region of a wide range of ingredients contained in industrial emissions [43]. This makes possible to determine a concentration of about a hundred ingredients along a laser beam, including carbon oxides (CO, CO<sub>2</sub>), nitrogen oxides (NO<sub>x</sub>), sulfur oxides (SO<sub>2</sub>, SO<sub>3</sub>), ozone (O<sub>3</sub>), ammonia (NH<sub>3</sub>), hydrosulfide (H<sub>2</sub>S), fluorine (F<sub>2</sub>), chlorine (Cl<sub>2</sub>), sulfur fluoride (SF<sub>6</sub>), methane (CH<sub>4</sub>), its derivatives and others. At the same time, the method ensures the accuracy of determination of a concentration to 0.01 of the maximum permissible concentration. The method almost does not lose sensitivity even at a distance of 5 km from a measuring point.

The main advantage of laser gas analysis is a possibility of creation of a remote, mobile system for studying zones of

small and large planes. Topographical objects or a volume of gas, which is irradiated because of backscattering, can act as reflectors of energy, which comes from a laser.

A basic equation of laser location appears on the basis of the dependence of the power radiation, which is reflected (scattered) by the object of zoning, at a certain wavelength from the distance to the object and its parameters [44]:

$$P_s = \frac{PS\eta\beta}{R^2} e^{2r}, \quad (1)$$

where  $P_s$  – is a power of the reflected (scattered) radiation;  $P$  – a power of laser;  $S$  – is an effective area of the receiving element;  $\eta$  – is efficiency of the receiving element;  $\beta$  is a volume backscattering coefficient of the incident radiation;  $\Gamma = \int_0^R \alpha(R) dR$  – integral coefficient of absorption of laser radiation;  $a$  – is a volume coefficient of absorption of laser radiation;  $R$  – is a distance to a zoning object.

Equation (1) of laser location requires to take into account features of radiation and scattering processes with subsequent capture and interpretation of the results obtained. Therefore, it is necessary to improve mathematical models for further use of a remote method to identify sources of unauthorized and uncontrolled emissions of pollutants into the air at technogenic sites, such as a waste landfill.

An important factor of the effectiveness of this method is its sensitivity, since it is necessary to identify the presence of an indicator substance at a low concentration accurately. The minimum concentration of contaminant admixture  $N_{\min}$ , which can be detected with a given error, characterizes the sensitivity of the differential absorption method [44, 45]. The specified error depends on characteristics of an optical signal.

To assess the sensitivity of a laser monitor, we should start from the relation

$$\ln P_s(\lambda) = \ln B - 2\Gamma(\lambda). \quad (2)$$

Then let us find the increment of the quantity  $T$ :

$$\delta\Gamma = -\frac{1}{2} \frac{\delta P_s}{P_s}.$$

Or for the module:

$$|\delta\Gamma| = \frac{1}{2} \frac{|\delta P_s|}{P_s}. \quad (3)$$

A signal/noise ratio  $Q$  determines the relative error in measuring  $P_s$  [46].

$$\delta = \frac{|\delta P_s|}{P_s} = \frac{1}{Q}.$$

One should measure  $P_s$  powers at two wavelengths and calculate their ratio in the differential absorption method. The relative error  $\delta$  doubles. Then the following ratio should determine the root-mean-square error in the estimate  $\Gamma$ :

$$\sigma_r = \frac{2}{Q}. \quad (4)$$

For example, for  $Q=10^5$  we have  $\sigma_r=2\cdot 10^{-5}$ .

For  $\delta\Gamma_{\min} = \sigma_r$  we have:

$$N_{\min} = \frac{\delta\Gamma_{\min}}{\Delta\sigma R_0} = \frac{\sigma_r}{\Delta\sigma R_0}.$$

For example, for  $R_0=5$  km,  $\Delta\sigma=3.7\cdot 10^{-23}$  m<sup>2</sup> we have:

$$N_{\min} = \frac{2\cdot 10^{-5}}{3.7\cdot 10^{-23} \cdot 5\cdot 10^3} \approx 10^{20} \text{ m}^{-3}.$$

Thus, with a background concentration of particles in the surface atmosphere  $N_b \approx 2\cdot 10^{25}$  m<sup>-3</sup>, we obtain  $N_{\min}/N_b \approx 2\cdot 10^{-6}$  or 5 ppm (particles per million air molecules). Such sensitivity of a laser monitor is sufficient for the diagnosis of contamination with inclusion of nanoparticles.

The main causes of errors are:

– laser complex equipment itself: laser frequency may not be stable, a laser measurement line has finite dimensions, radiation spectrum may be distorted, signal characteristics in the optical receiver may be distorted;

– a state of the atmosphere: fluctuations in atmospheric parameters, Doppler broadening of lines, shift of absorption lines, absorption by various interfering gases, presence of signals of purely rotational combinational scattering, fluctuations in gas temperature in the atmosphere.

We can eliminate the first group of sources by improvement of the quality of instruments of a laser complex.

We should pay special attention to a shift of absorption lines and absorption by various interfering gases when analyzing sources of errors associated with a state of the atmosphere. Corresponding errors can reach 1.6 and 2.6 %. At the same time, the total error does not exceed 3.0 %, and it is satisfactory for remote monitoring.

The obtained data and their comparison with literature sources [46, 47] make possible to assume that laser monitoring is suitable for the high-precision identification of gaseous substances in the atmosphere. However, there is a need to refine a mathematical model taking into account features of radiation and scattering processes, and to capture and interpret the results after.

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## 7. Conclusions

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1. Thinning of fibers of synthetic materials occurs as a result of a course of thermo-oxidative processes under the influence of the mass of waste. This makes possible to classify such materials as nanofibers.

2. Experimental studies on the example of a nitrene established gaseous substances, which are released during thermo-oxidative decomposition of polymers, and their concentration, depending on temperature of a process. There are substances-indicators of latent processes of combustion (smoldering) of materials in a body of a landfill. They are ammonia and hydrogen cyanide. In the future, it is necessary to simulate mentioned processes for a mixture of different materials in order to establish the dependence of a rate of development of thermo-oxidative processes in a mass of waste on conditions of their flow. It is important to determine an effect of nanostructures on thermo-oxidation processes of waste materials and a composition of released substances.

3. A frequency-tunable laser complex determines a concentration of substances for identification of gaseous products

of thermo-oxidative processes with high accuracy. The complex makes possible to determine a concentration of about one hundred of ingredients along a laser beam, among them are: carbon oxides (CO, CO<sub>2</sub>), nitrogen oxides (NO<sub>x</sub>), sulfur oxides (SO<sub>2</sub>, SO<sub>3</sub>), ozone (O<sub>3</sub>), ammonia (NH<sub>3</sub>), hydrosulfide (H<sub>2</sub>S), fluorine (F<sub>2</sub>), chlorine (Cl<sub>2</sub>), sulfur fluoride (SF<sub>6</sub>), methane (CH<sub>4</sub>), its derivatives and others. At the same time, the method provides the accuracy of determination of a concentration to 0.01 of the maximum permissible concentration. In this case, the laser location equation requires to take into account features of radiation and scattering processes, that is, there is a need for further improvement of mathematical models.

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