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Gas mixture composition control in fine organic synthesis

Fine organic synthesis includes a large number of stages. All its stages require determining the small quantities of impurities for initial and intermediate synthesis products. Fine organic synthesis is a complex process requiring automation. To automate the synthesis products' control device it is necessary to use the modern physico-chemical methods, which perform continuous measurements with high speed. This paper describes the hardware implementation of a device, based on infrared absorption analysis method. The absorption methods of substance composition analysis are based on the absorption of sounding radiation by the analysed component. The strength of the probing radiation passing through the mixture changes slightly at the small concentrations of the analysed component. Therefore, there is a problem of measuring small changes of a large signal. The absorption method of substance composition analysis can be used for analysis the composition of mixtures of gaseous organic substances. It enables to scan the optical frequency of the probe radiation by changing the angle of the interference filter. The proposed equipment allows determining the concentration of substances by overlapping their absorption spectra of infrared radiation.

Keywords: organic synthesis, analyzed gas, analysis of gas mixtures composition, infrared radiation (IR), spectrum of IR absorption coefficient, solid-state narrow-band interference optical filter, pyroelectric radiation receiver.

Introduction

Organic synthesis is a process of producing more complex organic substances from less complex organic or inorganic substances. People use the organic synthesis products extensively. Such products include: clothing, shoes, machine parts, toys, medicines, paints, dishes, etc.

There is basic and fine organic synthesis. Fine organic synthesis is the result of small-tonnage production of complex organic substances. Fine organic synthesis is based on a wide variety of chemical reactions, which are carried out by sophisticated equipment. Intermediate and by-products are formed at each stage of production. To improve the quality of complex production it is necessary to use the automatic control systems for production processes [1, 2].

Intermediate products of fine organic synthesis require fast and accurate control of their composition. These products may be solid, liquid or gaseous. The methods of analysis of solid and liquid composition enable the sampling of analyzed substance. These methods are well-studied and varied [3]. The methods of fast continuous analysis of gas mixtures composition are less diverse; therefore this work is devoted to their improvement.

Samples and Research Method

There are a lot of methods conducting the analysis of gas mixtures composition. Most of them have a high sensitivity to the non-measured components of the analyzed mixture, that is, they have a low selectivity of the measurement results. These methods include acoustic, ionization, diffusion, electrochemical methods as well as methods based on the thermal or ionization effects of the chemical reaction and changing the thermal conductivity of the analyzed mixture. These methods are effective in analyzing the mixture composition for more than two gases.

Fine organic synthesis often requires determining the composition of gas mixture, containing a large number of components. To solve this problem such methods as chromatography, mass spectroscopy and optical ones are used.

Chromatographic method of analysis of gas mixtures composition is based on separation the analyzed mixture's components due to effect of sorption. The separation of the gas mixture sample into components is carried out periodically in the chromatographic column. The disadvantages of this method include a low speed (it does not exceed two minutes), the necessity to use a carrier gas cylinder and to select a sorbent for each type of analyzed component.

Mass spectroscopy is based on separation of charged particles (usually ions) with different specific charges by magnetic field. This method uses pre-ionization of analyzed gas mixture. Complex, large and expensive equipment, including a vacuum pump must be used to apply this method.

Optical methods are based on the different abilities of the mixture components to interact with electromagnetic radiation of ultraviolet, visible and infrared (IR) wavelengths. Most gases do not interact with the visible wavelength range. Ultraviolet radiation mainly interacts with the atoms of gas molecules and it is used to determine the atomic composition of the analyzed mixture. To determine the molecular composition of gas mixtures, the most informative and universal methods are based on the interaction of the mixture components with infrared radiation.

There are several methods, which use the interaction of infrared radiation with gas molecules. They include the following:

1. Refractometric method based on the measurement of the refractive index of radiation. The measurement results of this method are characterized by low selectivity.
2. Methods based on laser scattering. They are mainly used for remote monitoring of atmospheric composition.
3. Methods based on the measurement of optical density, which are called absorption methods.

Each gas has its individual graph (spectrum) of IR absorption coefficient dependence from wavelength [4]. Therefore, absorption methods enable to determine selectively the quantity of components in the gas mixture. In the authors' view, absorption methods based on the measurement of IR absorption by a controlled component are quite universal and suitable for automation of thin organic synthesis.

The IR absorption method uses the characteristics of IR absorption spectra for different gases. The concentration of the controlled component of gas mixture is determined by the level of emission reduction that has passed through the analyzed mixture. Transmission the radiation with thickness l and frequency v by gas mixture is complied with the law of Buger-Lambert:

$$T_{\Gamma}(v) = \frac{I(v)}{I_0(v)} = \exp[-K(v)l] \quad (1)$$

$I_0(v)$ — the intensity of radiation before it passes the layer of the analyzed mixture; $I(v)$ — the intensity of radiation after it passes the layer of the analyzed mixture; $K(v)$ — gas mixture absorption coefficient for radiation with frequency v.

If the analyzed gas mixture contains L components, then according to Bayer's law, the absorption coefficient is described as:

$$K(v) = \sum_{j=1}^{L} C_j K_j(v) \quad (2)$$

$K_j(\nu)$ — radiation absorption coefficient for j component of the gas mixture; C_j — the concentration of the j component of the gas mixture.

The expressions (1) and (2) allow calculating the concentration of the j components, if the following conditions are met:

1. The selected probe radiation frequency (v_j) provides the ratio:

$$\max_{1 \le i \le L, i \ne j} K_j / K_i$$

Ideally, this condition means that only one j component of gas mixture absorbs the radiation at a frequency v_j.

2. The radiation absorption coefficient by the j component of gas mixture $K_j(\nu_j)$ at the frequency v_j is known.

3. The frequency v_j of radiation transmission by analyzed gas mixture is measured.

To measure the radiation transmission $T_\Gamma(v_j)$ by the gas mixture it is necessary to use the source of the probe radiation, the cuvette of the analyzed gas mixture's sample and the radiation receiver. The required frequency v_j of the probe radiation can be provided by the source of radiation with required frequency; by the radiation receiver, which is sensitive to radiation of a certain wavelength; by an additional spectral element, which enable to isolate the radiation of the required frequency.

The functions of infrared radiation transmission $T_\Gamma(v_j)$ can be described as a set of radiation absorption lines. The radiation absorption line for j gas with the center at the frequency v_j is characterized by the intensity of the absorption line S_{ji} of the j gas at the spectral interval i, the width δ_{ji} under normal conditions, the distance d_{ji} to the next line. The spectrum of radiation absorption by gas in the IR area of the spectrum consists of a set of closely located absorption lines. This set of lines is called a radiation absorption band.

The functions of radiation transmission by gases can be calculated by using the radiation absorption of specific frequency of each line of the spectrum. It is also possible to calculate the absorption function using the formulas of spectra models, or find it experimentally. The method of spectrum models is the most versatile; it has acceptable complexity of gas concentrations' calculations. This method includes the modeling of gas spectra with a set of absorption lines, suggesting a certain law of their location, intensity and width.

If the intensity of the absorption lines are described by exponential statistical law of distribution, and the distance between the lines and their width are described by uniform law of distribution, the IR absorption function for the mixture of L gases is calculated by the expression [5]:

$$T_{\Gamma_i} = \exp \frac{-l \sum_{j=1}^{L} S_{ji} P_j / d_{ji}}{\sqrt{1 + l \sum_{j=1}^{L} \left(S_{ji} P_j / d_{ji} \right) \Big/ \pi \sum_{j=1}^{L} \sum_{k=1}^{L} \left(\delta_{ji} P_k \sigma_{jki} / d_{ji} \right)}} \tag{3}$$

P_i — the concentration of the j gas in partial pressure units; σ_{jki} — the relative effectiveness of the optical collisions of j and k gas molecules; it characterizes the broadening of the absorption lines.

The expression (3) shows that the parameters of the absorption lines of infrared radiation depend on the composition of the gas mixture. In addition, the center of the absorption line v_j, its intensity S_{ji}, and width δ_{ji} depend on the temperature and pressure of the gas mixture. In measurements, if the frequency range is much more than the distance between the lines, the parameters of the radiation absorption band are more stable [5]. The allocation of such a frequency range can be provided by solid-state narrow-band interference optical filters [6].

It is difficult to calculate the concentrations P_j by using a well-known values of $T_{\Gamma i}$ in expression (3), because there is no effective mathematical methods enable to solve such systems of nonlinear equations and the obtained results may vary. Therefore, this expression must be simplified.

The research [5] shows that if the thickness of analyzed gas mixture is decreased and the components concentration P_j is small, the dependence of the absorption function on the components concentration can be described by linear absorption law:

$$T_{\Gamma_i} = 1 - l \sum_{j=1}^{L} \left(S_{ji} P_j / d_{ji} \right) \tag{4}$$

The functional diagram of the simplest device for measuring the concentration P_j of analyzed gas mixture is given in Fig. 1.

Figure 1. The functional diagram of the simplest device for measuring the gas concentration

In this illustration, the blocks have the following functions:
1 — broadband infrared source;
2 — solid-state narrow-band interference optical filter;
3 — cuvette with analyzed gas mixture;
4 — IR-to-electrical converter;
5 — electronic circuit for signal processing and displaying measurement results

The simplest scheme has a number of significant disadvantages. Firstly, non-measured gases in the analyzed mixture influence the component's concentration measurement only if they do not absorb IR radiation in the transparency range of the narrow-band interference optical filter. However, this condition is not always possible. Secondly, minimum concentration P_j of the analyzed component forms the maximum output voltage of the IR emitter to the electrical signal. For linear law of radiation absorption, described by expression (4), this means the necessity to measure small changes of a high signal.

If the source of broadband infrared radiation is made as a spiral, which is placed in the focus of parabolic mirror, then the thermal balance equation of the scheme (Fig. 1) for the emitter's spiral is:

$$q_{el} = q'_{tc} + q_{tc} + q_{con} + q_{rad} \quad (5)$$

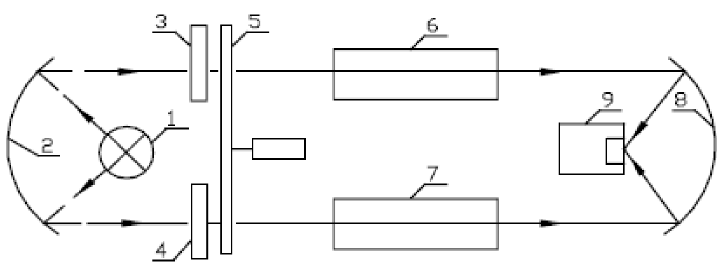
q_{el} — the energy, supplied to radiation source; q_{rad} — the energy, which creates infrared radiation; q_{tc} — the energy, which heats the device body by thermal conductivity in air; q'_{tc} — thermal conductivity through the current-carrying contacts; q_{con} — convective heat exchange.

The analysis of expression (5) shows that the power of probe radiation depends on the details of the scheme (Fig. 1) significantly.

The ambient temperature increase, the power of IR radiation also increases. The step value is approximately (0.3 — 0.4) * 10^{-3} watt*Kelvin^{-1}; it is 31 % in the range from -20 ˚C to +30 ˚C. The degree of deviation from linear dependence is up to 4.6 %. For these measurement errors, the linear absorption law described by expression (4) cannot be applied. The measurement of small concentrations of analyzed components is not achievable.

To reduce the dependence of the infrared radiation power on the ambient temperature by the minimum threshold of the determined concentration, the scheme (Fig. 1) is complemented by additional (reference) channel. This reference channel contains a cuvette. It is filled with a reference gas mixture, which includes the identical analyzed gas mixture, but without analyzed component. The analytical signal is the difference in the IR flow that passed through the main and additional cuvettes. These cuvettes are filled with analyzed and reference gas mixtures. To calculate the difference of IR flows, it is necessary to put them one after another to the converter of IR signal to electrical signal. The order of IR flow is provided by a mechanical modulator.

A typical scheme of gas mixture component concentration measurement with a mechanical modulator of probe radiation, main and additional reference measuring channels is shown on Fig. 2.



(1 — source of radiation; 2 — parabolic radiation flow detector; 3 and 4 — solid-state narrow-band interference optical filters; 5 — mechanical modulator; 6 — working cuvette; 7 — reference cuvette; 8 — focusing system; 9 — converter of IR signal into electrical signal).

Figure 2. Differential symmetric scheme of the device, which implements the absorption spectral method for measuring the gases concentration

In this scheme, the pyroelectric radiation receiver is used as converter of IR signal into electrical signal [7]. This type of converter, for example, the IRA-E410S1, is characterized by high sensitivity and a wide (from 2 to 20 microns) wavelength range of perceived IR radiation. The receiver only responds to changes in IR radiation, therefore it is necessary to use the probe radiation modulator. The electrical signal from the receiver output is AC signal. This allows applying the simultaneous detection for signal processing to further reduce measurement errors.

The scheme presented in Fig. 2 has a number of disadvantages. Firstly, the additional reference channel complicates the scheme of the measurement. Each additional element has changing over time parameters and is the source of additional error of measurement results. Secondly, the additional reference cuvette *7* cannot be completely identical to the working cuvette *6* in parameters and external effects. The reference cuvette *7* must be well sealed while the analyzed gas mixture is passed through the working cuvette. Therefore, it is difficult to achieve the same temperature of these elements of the measurement device. In addition, the walls and windows of working cuvette are more vulnerable to contamination. All these processes create a difference in IR flows on the outputs of cuvettes *6* and *7* when they do not have a measured component of the analyzed mixture. As a result, the minimum detected concentration of the analyzed gas is increased. Thirdly, the use of a mechanical modulator causes a number of specific interference. For example, interference caused by the mechanical luff of the modulator parts.

Results and Discussion

This article proposes a measuring scheme without above-mentioned disadvantages. The main idea of the scheme includes the modulation the optical frequency of probe IR. To implement the absorption spectral method for gas concentration measurement, the solid-state narrowband interference optical filters are used. Such filters determine the required spectral interval of the probe radiation. The solid state narrowband interference optical filters are designed as a set of transparent films, which are placed at a transparent for IR radiation substrate. These films alternate large and small values of refractive parameters [8]. The optical frequency of the emitted radiation depends on the thickness of films placed at the substrate. When the radiation decrease's angle on the filter is changed, its spectral characteristics (the dependence of the transmission *T* on the wavelength λ) also are changed. This dependence is shown on Fig. 3 [8].

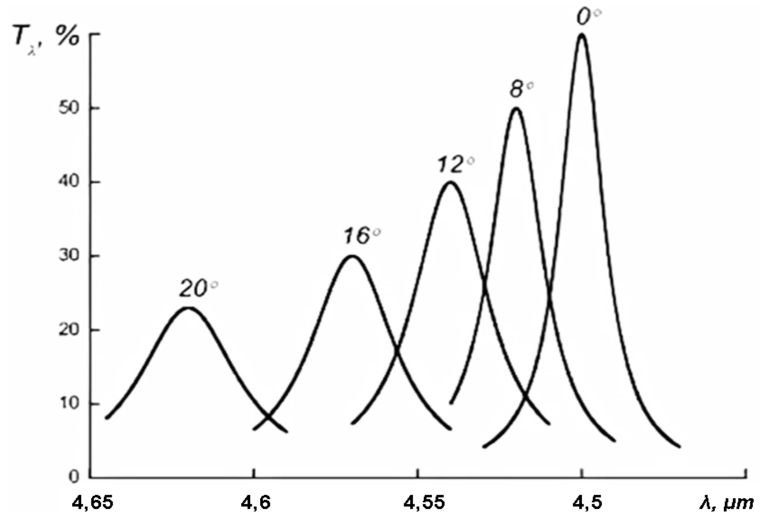


Figure 3. The dependence of the filter's spectral light transmission on the radiation decrease's angle

The work of the scheme will be discussed by measuring the carbon monoxide concentration in gas mixture. This gas is widely used in organic synthesis.

It is proposed to form a beam of parallel rays of a wide-range IR. This beam of rays is passed through a narrow-band interference optical filter at an angle that changes according to the sinusoidal law. For example, using a filter similar to shown on Fig.3 and changing the angle from 0 to 16°, the IR wavelength, which corresponds to the maximum filter transmission, will vary from 4.5 to 4.67 μm.

Fig. 4 shows the dependence of radiation absorption by carbon monoxide on the IR wavelength. The figure presents that the IR of 4.5 μm is weakly absorbed by carbon monoxide. The maximum of IR absorption corresponds to a wavelength of 4.67 μm.

It is proposed to pass IR flow, modulated by filter on optical frequency, through the analyzed gas mixture. The intensity of the passed radiation through the gas mixture is monitored by the IR receiver. If carbon monoxide is not present in the analyzed gas mixture, there should be no output signal of the receiver. The appearance of carbon monoxide in the analyzed gas mixture causes output signal of the receiver. The amplitude of this signal is proportional to low concentrations of carbon monoxide.

The Fig. 5 presents the implementation scheme of the absorption method of gas mixture component's concentration measurement by modulating the optical frequency of IR.

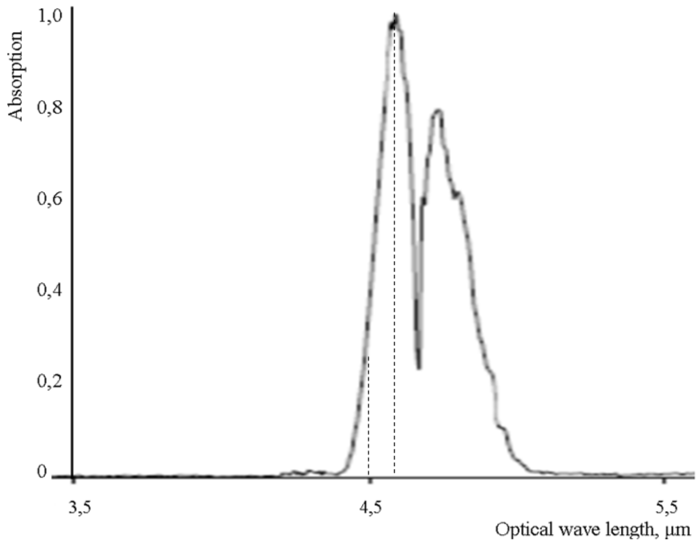


Figure 4. The dependence of radiation absorption by carbon monoxide on the frequency of infrared radiation

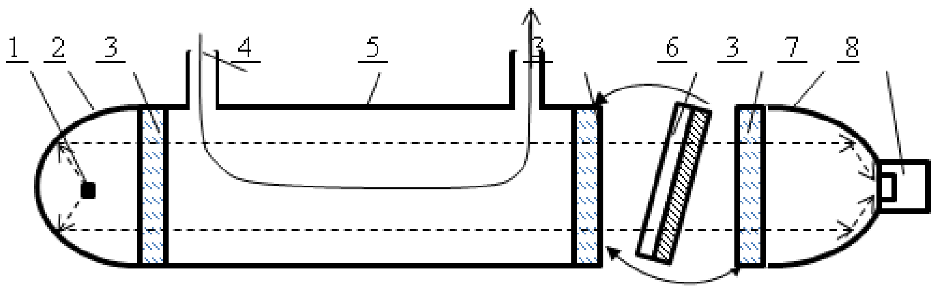


Figure 5. Implementation scheme of the absorption method of gas mixture component's concentration measurement by modulating the optical frequency of IR

The scheme contains the following elements: a broadband IR source (1), made as a spiral placed in the focus of a parabolic mirror (2); windows made of fluorite CaF_2, which is transparent for IR material (3); cuvette (5) with analyzed gas mixture (4); solid-state narrow-band interference optical filter (6) with periodically changing installation angle; parabolic mirror (7), focusing radiation on the radiation receiver (8).

The used in this scheme pyroelectric radiation receiver IRA-E410S1 [7] is sensitive only to the signal, which is variable in time. The examination of this receiver shows that it has the highest sensitivity if the IR flow is changed in time with a frequency of about 7 Hz. Therefore, the mechanical drive for changing the angle of interference optical filter installation operates at a frequency of 7 Hz.

A study of scheme (Fig. 5) showed that if there is no carbon monoxide in the analyzed mixture (4), there is an output signal of the receiver (8). This can be explained by the fact that when the radiation decrease's angle to the narrow-band interference optical filter is deviated from 90°, the frequency of passed infrared radiation is decreased, the coefficient of light transmission of the filter is decreased and the width of the spectral range of the passed radiation is increased. Therefore, the changing the angle of the filter may cause increase as well as decrease the intensity of radiation passed through the IR filter. Modulation radiation appears. This phenomenon reduces the effectiveness of proposed method significantly.

To reduce the dependence of the intensity of the radiation passing through the IR filter on the installation angle of this filter, a compensating device is proposed [8–10]. It is a disk connected to the IR filter at an angle φ. This angle is equal to the maximum angle of the IR filter installation and can vary from -φ to +φ periodically. The disk of compensating device contains a hole; its diameter is equal to the beam diameter of the infrared radiation filter. A scheme of connection the compensating device and the IR filter is presented at Figure 6. The IR filter (1) is placed in the mandrel (2), which makes rotational — oscillating movements at an angle of φ. The IR filter is attached by a nut (3). This nut is fitted with a compensating device (4). It is adjusted by rotating in relation to the IR filter. The adjusted compensation device is attached by a nut (5). Testing the scheme presented at figure 5, it was possible to reduce the modulation radiation by 16.5 times.

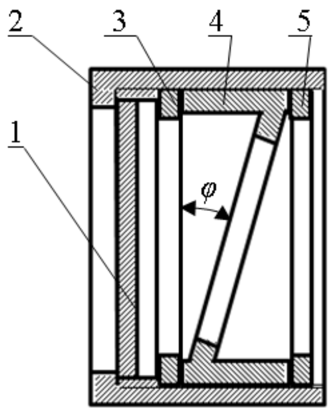


Figure 6. Connection the compensating device and the IR filter

Further analysis the influence of ambient temperature's change on output signal of the receiver (8) has revealed the effect of mirror reflection by the reverse side of the IR filter from the surface of the parabolic mirror on the surface of the receiver. To reduce this effect, the receiver window is replaced by a band interference filter that passes the radiation from 4.3 to 5.4 μm. These changes are presented in Fig. 7.

The final implementation scheme of the absorption method for gas concentration measurement in gas mixture by modulating the optical frequency of IR is shown in Fig. 7.

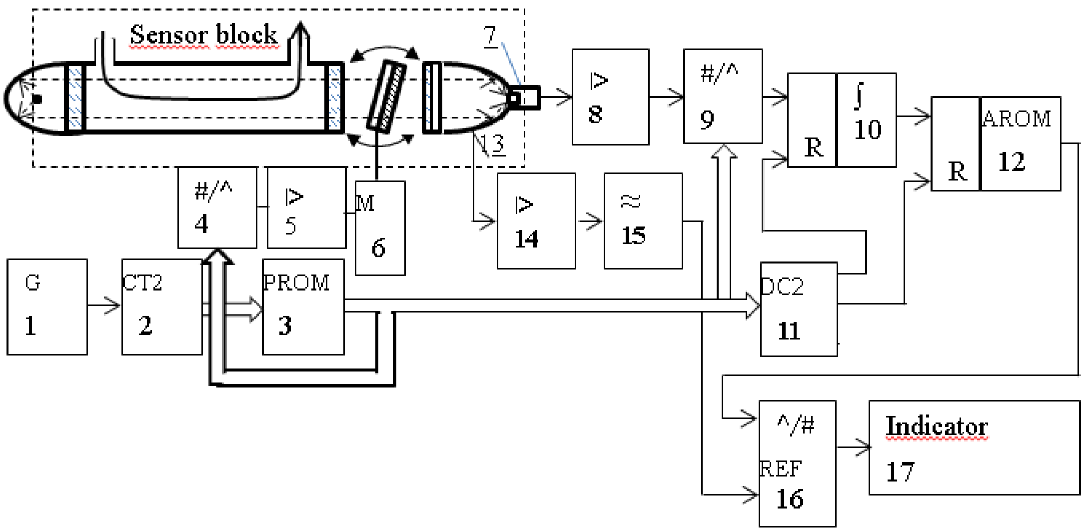


Figure 7. Functional implementation scheme of the absorption method for gas concentration measurement in gas mixture by modulating the optical frequency of IR

The proposed scheme has several blocks. The generator (1) generates pulses that are delivered to a binary counter (2). The binary code from the counter goes to the address inputs of the programmable read-only memory device (3). This device contains the sinusoidal function codes. These codes are converted to analogue signal by a digital analogue converter (4), amplified by the amplifier (5) and change the installation angle of the interference IR filter according to the sinusoidal law by electromechanical drive (6).

The variable component of the analyzed gas mixture passed through the radiation is perceived by the receiver (7). The output signal from the receiver is amplified by the amplifier (8). Then to determine the amplitude of the variable signal accurately, synchronous detection operation is performed. The signal is multiplied by a sinusoidal function using a multiplying digital-to-analogue converter (9). The output signal of the converter is integrated by the integrated amplifier circuit (10) for 8 periods and is stored by the sample and storage device of the analogue signal (12). The decipher (11) carries out the synchronization of the reset commands of the integrated amplifier and the signal memory. The output analogue signal of the sampling and storage device (12) is proportional to the measured concentration of carbon oxide [11–13].

The constant component of the radiation, passed through the analyzed gas mixture, is perceived by the receiver (13). The compensated thermal elements TK-1 × 1.5 made on antimony-bismuth junctures are used as a receiver. The output signal of the receiver (13) is amplified by the amplifier (14) and goes to the low-pass filter (15).

To reduce the influence of cuvette window contamination and radiation intensity I_0 before passing through the gas mixture on the measurement results it is necessary to apply a signal division operation from the sample and storage device output (12) to the low pass filter output (15) [14–16]. The division operation is performed by analogue-digital converter (16). The results are displayed digitally by an indicator (17).

The dependence of the output signal U on time t at low concentrations C of the measured gas is described by the expression:

$$U(t) = K_R IT_{OS}\left[k\tau_m(\varphi(t))\delta_{0.5}(\varphi(t)) - C\int_0^\infty lK(\gamma)\tau_F(\varphi(t))d\gamma\right] = K_R IT_{OS}\left[A(t) - CB(t)\right] \quad (6)$$

K_R — IR conversion coefficient of receiver; I_0 — radiation intensity before passing through gas mixture; T_{OS} — radiation transmission function by optical system; k — coefficient of signal's variable component attenuation by compensation device (Figure 6); τ_m, φ, $\delta_{0.5}$ — maximum transmission, angle of installation and half width of IR filter; $K(\gamma)$ — the spectral characteristic of analyzed gas; $\tau_F(\gamma)$ — the spectral characteristic of the IR filter; γ — the optical frequency [17].

The test results of the scheme, implementing the absorption method of carbon monoxide concentration measurement in the air by modulating the optical frequency of IR radiation, are presented in Table 1.

Table 1

Test results of proposed method

Concentration of carbon oxide, ppm		Error
Absorption method, C_A	Chemical method, C_{CH}	$\Delta = 100(C_{CH} — C_A) / C_{CH}$, %
187	198	5.5
94	102	7.8
45	56	19.6
12	23	47.8

The table 1 shows that obtained results revealed satisfactory precision of measurement method.

Conclusion

The obtained results showed that the proposed equipment of implementation the absorption method for gas concentration measurement in gas mixture by modulating the optical frequency of IR reveals satisfactory precision with recognized chemical measuring methods. At the same time, the proposed equipment provides faster measurement time, which does not exceed one second. Therefore, the proposed equipment for gas components control is recommended to use in thin organic synthesis.

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Жұқа органикалық синтезде газтәрізді компоненттердің құрамын бақылау

Жұқа органикалық синтез көптеген кезеңдерді қамтиды. Ол әр кезеңде бастапқы және аралық синтез өнімдерінің қоспаларының аз мөлшерін анықтауды талап етеді. Жұқа органикалық синтез — бұл автоматтандыруды қажет ететін күрделі процесс. Синтез өнімдерінің құрамын бақылау құрылғыларын автоматтандыру жоғары жылдамдықпен үздіксіз өлшеуді жүргізуге мүмкіндік беретін заманауи физика-химиялық әдістерді қолдануды қажет етеді. Газтәрізді органикалық заттардың күрделі қоспаларының құрамын инфрақызыл сіңіруді талдау әдісін қолданатын техникалық жабдықты аппараттық түрде енгізу ұсынылған. Заттардың құрамын талдауға арналған сіңіру әдістері зерттелетін компонент бойынша зонд сәулесінің сіңуіне негізделген. Зерттелетін қоспа арқылы берілетін зонд сәулесінің қуаты, зерттелетін компоненттің төмен концентрациясында аз мөлшерде өзгереді. Сондықтан үлкен сигналдағы кішкентай өзгерістерді өлшеу кезінде мәселе туындайды. Адсорбциялық талдау әдістерін құрамында органикалық заттары бар газ қоспаларының құрамын талдау үшін қолдануға болады. Әдістің ерекшелігі — зонд сәулесінің оптикалық жиілігін интерференциялық фильтрдің бұрышын өзгерту арқылы сканерлеу. Ұсынылған жабдық сізге инфрақызыл сәулеленудің жұтылу спектрлерін қабаттастыру арқылы заттардың концентрациясын анықтауға мүмкіндік береді.

Кілт сөздер: органикалық синтез, талданған үлгі, газ қоспаларының құрамын талдау, инфрақызыл сәуле, инфрақызыл сәулеленудің жұтылу коэффициентінің спектрі, қатты денелі тар диапазондағы кедергі оптикалық сүзгі, пироэлектрлік сәулелену детекторы.

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Контроль состава газообразных компонентов при тонком органическом синтезе

Тонкий органический синтез содержит большое число стадий. Он требует на каждой стадии определения содержания малых количеств примесей исходных и промежуточных продуктов синтеза. Тонкий органический синтез является сложным технологическим процессом, требующим автоматизации. Автоматизация устройств контроля состава продуктов синтеза реализуется путем применения современных физико-химических методов, позволяющих проводить непрерывные измерения с высоким быстродействием. Авторами предложено аппаратурное распределение технических средств, реализующих метод инфракрасного абсорбционного анализа состава сложных смесей газообразных органических веществ. Абсорбционные методы анализа состава веществ основаны на поглощении зондирующего излучения анализируемым компонентом. Мощность зондирующего излучения, прошедшего через анализируемую смесь, при малых концентрациях анализируемого компонента изменяется на незначительную величину. Поэтому возникает задача измерения малых изменений большого сигнала. Адсорбционные методы анализа могут использоваться для анализа состава газовых смесей, содержащих органические вещества. Особенностью метода является сканирование оптической частоты зондирующего излучения путем изменения угла установки интерференционного фильтра. Предложенная аппаратура позволяет определять концентрации веществ при перекрывании их спектров поглощения инфракрасного излучения.

Ключевые слова: органический синтез, анализируемая проба, анализ состава газовых смесей, инфракрасное излучение, спектр коэффициента поглощения ИК излучения, твердотельный узкополосный интерференционный оптический фильтр, пироэлектрический приемник излучения.

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